30 YEAR OPERATION AND MAINTENANCE PLAN VOLUME -3 SAMPLING AND ANALYSIS PLAN

PART II QUALITY ASSURANCE PROJECT PLAN

ASBESTOS DUMP SUPERFUND SITE OPERABLE UNIT NO. 1 MILLINGTON, NEW JERSEY

Submitted to:

New Jersey Department of Environmental Protection
Division of Hazardous Site Mitigation
Hazardous Waste Programs
Trenton, New Jersey

United States Environmental Protection Agency Region II New Jersey Remediation Branch New York, New York

U. S. Army Corps of Engineers
New York District
Environmental Residency Northern New Jersey Area
East Brunswick, New Jersey
Submitted by:



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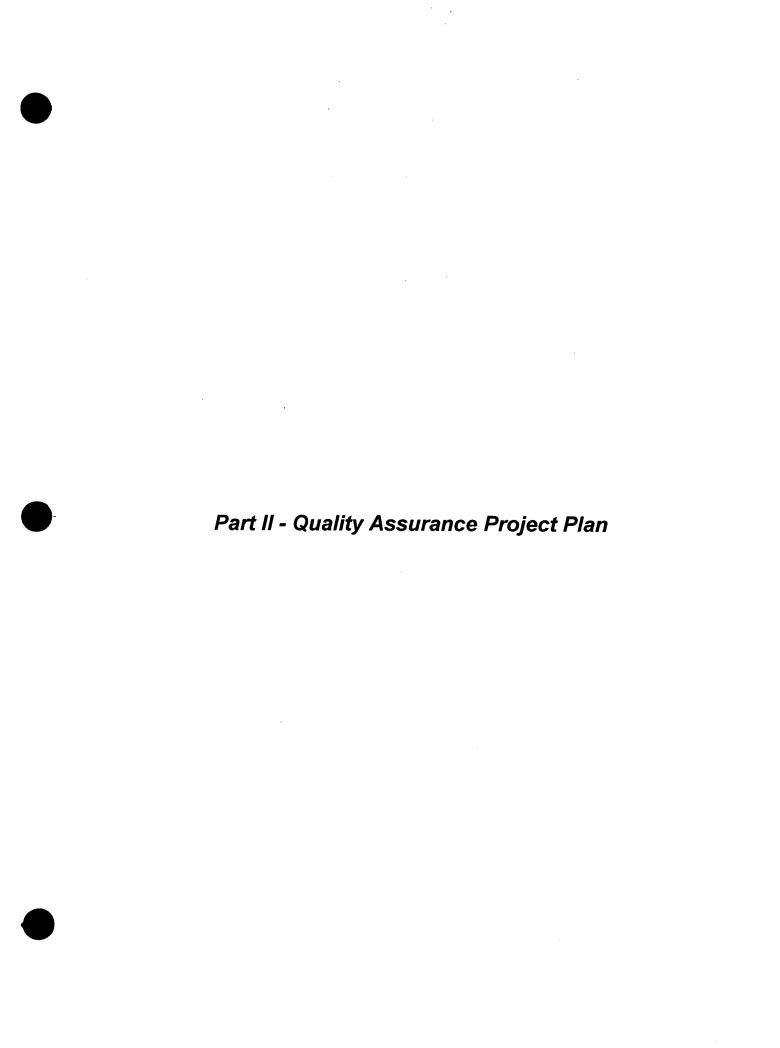


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List of Acronyms

AALA American Association for Laboratory Accreditation

AR/COC Analysis Request / Chain of Custody

ASCII American Standard Code Information Interchange

ASTM American Society for Testing and Materials

°C degrees Celsius

CAR Corrective Action Report

CCB continuing calibration blank

CCQC Contractor Chemical Quality Control

CVC continuing calibration verification

CERCLA Comprehensive Environmental Response, Compensation, and Liability Act

CF calibration factor

CFR Code of Federal Regulation

CIH Certified Industrial Hygienist

CL control limit

CLP Contract Laboratory Program

COC chain of custody

CQCM Contractor Quality Control Manager

CQCP Contractor Quality Control Plan

CX Center of Expertise

DCQCR Daily Chemical Quality Control Reports

DOT Department of Transportation
DQCR Daily Quality Control Report

DQI data quality indicator
DQO data quality objective

EDD electronic data deliverable EICP extracted ion current profile

EPA Environmental Protection Agency

FADL Field Activity Daily Log

FS Feasibility Study
FSP Field Sampling Plan
FWV Field Work Variance

g gram

G glass

GC/MS gas chromatography/ mass spectroscopy

HCl hydrochloric acid

HNO₃ nitric acid

HPLC high-performance liquid chromatography
HTRW Hazardous, Toxic, and Radioactive Waste

ICB initial calibration blank

ICP inductively coupled plasma

ICPAES Inductively Coupled Plasma Atomic Emission Spectroscopy
ICPOES Inductively Coupled Plasma Optical Emission Spectroscopy

ICS interference check standard ICV initial calibration verification

ID identification

IDW investigation-derived waste

IT IT Corporation

ITEMS International Technology Environmental Management System

LCL lower control limit

LCS laboratory control sample

LOR Letter-of-Receipt

MCLG Maximum Contaminant Limit Goal

MDL method detection limit mg/kg milligrams per kilogram

mg/L milligrams per liter

mL milliliter

MS matrix spike

MSD matrix spike duplicate

N/A not applicable

NCR Nonconformance Report

NIST National Institute of Standards and Technology

NJDEP New Jersey Department of Environmental Protection

NPDES National Pollutant Discharge Elimination System

NPL National Priority List (Superfund)

OU operable unit
P polyethylene

PARCCS Precision, accuracy, representativeness, completeness, comparability and

sensitivity

PC project chemist

PE performance evaluation

PM Project Manager

PMO Program Management Office

ppb parts per billion ppm parts per million QA quality assurance

QAM quality assurance manager

QAP quality assurance plan

QAPP Quality Assurance Project Plan

QC quality control

QCSR Quality Control Summary Report

R recovery

RCRA Resource Conservation and Recovery Act

RF response factor

RFP Request for Proposal
RI Remedial Investigation

RL reporting limit

RPD relative percent difference

RRF relative response factor

RSD relative standard deviation

SAP Sampling and Analysis Plan

SI Site Investigation

SOP standard operating procedure

TO Task Order

TPP Technical Project Planning

UCL upper control limit

USACE U.S. Army Corps of Engineers
USDA U.S. Department of Agricultural

WP work plan

μg/kg microgram(s) per kilogram

μg/L microgram(s) per liter

 $\mu g/mL$ microgram(s) per milliliter

 μL

microliter(s)

1.0 Project Description

This portion of the Sampling and Analysis Plan (SAP) consists of the Quality Assurance Project Plan (QAPP). It will be used to control all analytical activities during the monitoring period at the Millington Asbestos Dump OU-1 site located in Millington, New Jersey (Figure 1-1, Site Layout Drawing, of the Field Sampling Plan (FSP)). These activities will be performed during the 30-year post-closure monitoring period managed by the New Jersey Department of Environmental Protection (NJDEP) and the United States Environmental Protection Agency (USEPA). The USEPA requires that all environmental monitoring and measurement efforts mandated or supported by these organizations, participate in a centrally managed quality assurance (QA) program. Any party generating data for this project has the responsibility to implement procedures to ensure that the precision, accuracy, representativeness, and completeness of the data are known and documented. To ensure these responsibilities are met uniformly, each party must adhere to this QAPP.

This QAPP presents the organization, objectives, functional activities, and specific QA and quality control (QC) activities associated with the SAP for the OU-1 investigations. It describes the specific protocols that will be followed for sample handling and storage, chain of custody, and laboratory analyses. This plan also presents details regarding data quality objectives for the project, sampling and preservation procedures for samples collected in the field, sample documentation, sample packaging and shipping, and laboratory analytical procedures for all media sampled.

All QA/QC procedures will be in accordance with applicable professional technical standards, EPA and USACE requirements, government regulations and guidelines, and specific project goals and requirements. This QAPP was prepared by the IT Corporation (IT) in accordance with the following EPA QAPP and USACE guidance documents: QA/G-5, EPA Guidance For QAPPs (EPA 1998); Interim Final QA/R-5, EPA Requirements For QAPPs For Environmental Data Operations (EPA 1999); Chemical Data Quality Management for Hazardous, Toxic, Radioactive Waste Remedial Activities (USACE EM-1110-1-623, 1998); Requirements for the Preparation of Sampling Analysis Plans (USACE EM 200-1-3, 1994a); and EM-200-1-6, Chemical Quality Assurance for HTRW Projects (USACE 10 Oct 1997). Field QA/QC and sampling procedures will be updated as appropriate to incorporate changes pertinent to the Millington SOW that may be published in revisions to the guidance documents referenced above.

Chapter 1.0 of the FSP portion of this SAP contains the project description and the site history. The FSP also contains the detailed discussion of the sampling methods.

2.0 Project Organization and Responsibilities

The discussion regarding the general project organization and responsibilities has been provided in Chapter 2.0 of the FSP. The FSP provides the current general project organization complete with lines of authority within the project and program organization. The NJDEP/USEPA, the designated contractor (currently IT subject to revision by NJDEP), teaming members, and subcontractor positions, which have responsibility for obtaining analytical data for the project are disscussed in the subject document. The information presented within provides the organization and responsibilities of the environmental laboratories that will provide analytical services under the contract. NJDEP at their discretion may designate other contractors and labs for completion of the subject tasks.

2.1 Subcontractor Analytical Laboratories

Analytical laboratory support specific to the OU-1 investigations will be obtained from various independent laboratories. The analytical services will be designated to subcontractors based on their capacities and capabilities. The selected subcontract laboratory shall possess New Jersey Department of Environmental Protection (NJDEP) certification or have certification from a state, which has reciprocity with NJDEP's certification program such as NYDOH). The chosen subcontractor assumes the responsibilities to assure the NJDEP/USEPA that the subcontracted certified laboratories meet the operational requirement of certification and that their certification is maintained throughout the lifetime of the project. Relevant QA Manuals, laboratory qualification statements, certifications, and license documentation have been provided in Appendix B of this manual.

Organization charts presenting key laboratory personnel and organization will be provided in the chosen labs Quality Assurance Plan (QAP). The responsibilities of key personnel are described in the following paragraphs. The assignment of personnel to each position will be based on a combination of (1) experience in the type of work being performed, (2) experience working with NJDEP/USEPA personnel and procedures, and (3) a demonstrated commitment to high quality and timely job performance.

Prior to commencement of field activities for the project, the contractor should provide a complete copy of the OMP and this QAPP to the chosen laboratory for review.

2.2 Analytical Laboratory Organization

The following subsections present the general organization for the analytical laboratory. Additional details are presented in Section 4.0 of the laboratory's QAP.

2.2.1 Laboratory Quality Assurance Manager (QAM)

The subcontractor Laboratory QAM is responsible for the laboratory QA/QC in accordance with the requirements of this QAPP and in conjunction with the laboratory's established laboratory QA Program. In coordination with the Project Chemist, this individual will be responsible for documentation of the following:

- samples received by the laboratory were analyzed in accordance with required methodologies
- instrument calibrations were performed properly and documented
- field and internal laboratory QC samples were analyzed and documented
- all analytical results for both field and QC samples were reported to the contractor in an acceptable electronic format capable of being transmitted into a database.

The QAM is also responsible for processing laboratory Non-Conformance Reports (NCRs) in a timely manner and for implementing Corrective Action Report recommendations and requirements. The laboratory's QAM reports directly to the Project Chemist for issues related to this project.

2.2.2 Laboratory Project Manager

It will be the responsibility of the analytical laboratory to assign one Project Manager (and backup) who will be the Prime Contractor's (which will be designated by NJDEP) single point of contact. The Laboratory Project Manager will be responsible for:

- initiation and maintenance of the services contract with IT on individual job tasks
- preparation of all laboratory-associated work plans, schedules, and manpower allocations
- provision of day-to-day direction of the laboratory project team including analytical department managers, supervisors, QA personnel, and data management personnel
- coordination of all laboratory related financial and contractual aspects of the project
- provision of formatting and technical review for all laboratory reports
- provision of day-to-day communication with the contractor; provision of final review
- approval on all laboratory analytical reports to the contractor

response to all post-project inquiries.

If at all possible the laboratory project manager will be assigned for the duration of the project.

2.2.3 Laboratory Director

The Laboratory Director shall have complete authority for the overall analytical laboratory operations. The responsibilities of the analytical Laboratory Director include the following:

- coordination of all analytical production activities conducted within the analytical departments
- working with the Laboratory Project Manager to ensure all project objectives are met
- provision of guidance to analytical department managers
- facilitation of transfer of data produced by the analytical departments to the report preparation and review staff for final delivery to the client.

If at all possible the laboratory director will be assigned for the duration of the project.

2.2.4 Laboratory Analysis Team Leaders

The responsibilities of each Analysis Team Leader include the following:

- coordination of all analytical functions related to their specific analytical areas
- provision of technical information to and oversight of all analysis being performed
- review and approval of all analytical results produced by their specific analytical area of expertise
- maintenance of all analytical records and information pertaining to the analysis being performed.

If at all possible the laboratory analysis team leader will be assigned for the duration of the project.

2.2.5 Laboratory Staff Chemists and Technicians

The responsibilities of the Staff Chemists and Technicians include the following:

- perform assignments made by the their respective team leaders
- maintenance, repair, and calibration of equipment assigned to them
- extraction, digestion, cleanup and preparation of samples extracts for instrumental analysis
- sample analysis and review of the work accomplished

If at all possible the laboratory chemists and technicians will be assigned for the duration of the project.

2.2.6 Laboratory Sample Management Team Leader

The Sample Management Team Leader shall be responsible for the receipt of all environmental samples and handling them in such a manner that the external and internal chains of custody are not violated. This individual is responsible for the handling, control, inspection, safekeeping and disposal of all environmental samples following receipt by the analytical laboratory. Additionally, this person is responsible for defining sample supply requirements and providing them to the client in a timely manner.

2.2.7 Laboratory Data Management Team Leader

The laboratory shall maintain a full time Data Management Team Leader, or equivalent, whose responsibilities include: support and maintenance of the laboratory database; initiate the creation of ITEMS compatible electronic data; and serve as the single point-of-contact for transmission of the electronic data deliverables and corrections of versions with problems.

2.2.8 Technical Backup for All Positions

The analytical laboratory shall perform sufficient support training of backup personnel, so that there are no instances whereby vacations, illness, or excused absences interfere with the acceptable handling and turnaround of the contractor's environmental samples.

3.0 Data Quality Objectives

Data Quality Objectives (DQOs) are qualitative and quantitative statements derived from the DQO process that specify, from an end user's perspective, the quality of data required to support decisions made during investigative and/or remedial activities. The DQOs specify the maximum level of uncertainty the user is willing to accept, while not affecting the accuracy of project decisions. DQOs are developed prior to data collection and should be specified for all data collection activities that take place.

3.1 Project Objectives

Specifically, the overall project objectives with respect to data quality are to obtain analytical data, which are technically sound and legally defensible. This is to be accomplished through the proper implementation of the field sampling procedures, chain of custody (COC) documentation, controlled laboratory analysis, and review or validation of the reported data prior to their use. General project objectives are:

- Provide data of sufficient quality and quantity to support the ongoing remedial investigation and feasibility studies.
- Obtain groundwater, surface water, and sediment data that can be compared to NJDEP and Region II EPA Federal standards (i.e. analytical reporting limits for groundwater must be lower than the Maximum Contaminant Limit Goal (MCLG) of 7,000,000 fibers per liter.)
- Ensure samples are collected and analyzed in accordance with the approved procedures established within this document.
- Specify the necessary QA/QC procedures for all environmental activities to meet USACE and other applicable agency requirements.

The necessary procedures for field sampling, COC, laboratory analysis, reporting of data and corrective actions are discussed in other sections of this QAPP.

3.2 Data Quality Design Process

The USACE data quality design process is basically a four-phase process performed by a technical planning team to identify the data needed to support specific project decisions and to create a data collection program capable of collecting the necessary data. The DQOs, generated

as a result of the TPP process, meet the USACE definition of DQO and are project-specific statements that include the nine data quality requirements:

- Project objective(s) satisfied
- Data user perspective(s) satisfied
- Contaminant or characteristic of interest identified
- Media of interest identified
- Required sampling areas or locations and depths identified
- Number of samples required
- Reference concentration of interest or other performance criteria identified
- Sampling method identified
- Analytical method identified.

A complete description of this process can be found in the USACE Engineering Manual, EM-200 1-2, *Technical Project Planning (TPP) Process* (USACE)

3.2.1 Identify Current Project Strategy

The current implemented strategy at the Millington Asbestos Dump OU-1 Superfund Site has been presented in Chapter 1.0 of the FSP and will be summarized here. Currently, the contamination is considered encapsulated with in the confines of OU-1. This area will be sampled for Asbestos Containing Materials in water and sediment on a staggered sampling schedule to ensure that the final remedy for this area is functioning as designed.

3.2.2 Determine Data Needs

This step of the technical planning process provides the data requirements for two general categories: data needs from a remedy perspective to support implementation of the remedy at the site and data needs from a compliance perspective to satisfy applicable state and federal requirements. The data quality must take the following information into account:

- data needed to provide long term monitoring of the effectiveness of the overall remedial objective, encapsulation of asbestos and asbestos fibers by characterizing the extent of migration of asbestos off-site
- data user to include the USACE and USEPA
- intended use of data including long term monitoring and comparison to MCLs and background concentrations
- number of samples necessary to determine effectiveness of the remedial objective
- reference concentration of asbestos 7,000,000 fibers per liter MCL in groundwater
- Area of interest or desired sampling location(s) and depth(s).

3.2.3 Develop Data Collection Options

The next phase of the TPP process is to design and plan the sampling and analysis activities necessary to fulfill the data needs. Basic data collection was chosen since there is only the current project objectives to fulfill. The following must be taken into the procedure steps:

- Data needs are to provide long term monitoring of the effectiveness of the overall remedial objective, encapsulation of asbestos and asbestos fibers by comparing results to MCL and background values
- The project objectives to be satisfied are in support of the overall remedial objective, encapsulation of asbestos and asbestos fibers
- Number of samples that are to be collected
- Locations from where the samples are to be collected based on existing monitoring wells and cap boundaries
- Sample collection methods to be used for water and sediment
- Sample analysis methods to be used for asbestos in water and sediment
- List limitations, benefits or requirements associated with each data collection option.

3.2.4 Finalize Data Collection Program

This final step of the DQO process is to create a sample collection program that best fits the long-term and short-term goals of the project. The finalized data collection program is provided in the FSP portion of this SAP.

3.3 Quality Assurance Indicators for Analytical Data

The final step in establishing the data quality objectives is to prepare the analytical data quality indicators (DQIs), know as the precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS) parameters. The DQI summaries are found in Tables 3-1 and 3-2 and a further discussion of these parameters can be found in Chapter 9.0 of this QAPP. Once the analytical laboratory is chosen to perform the analytical work, they will provide their laboratory quality assurance plan. The laboratory QAP shall include the Standard Operating Procedures (SOPs) and in-house generated quality control limits for all parameters that they have been contracted for and have agreed to determine

3.3.1 Precision

Precision is determined and reported as the relative percent difference (RPD) between the results for field duplicates and/or between the results for the spiked duplicate control samples (MS/MSD). Data with acceptable quality shall meet the precision criteria established in the site-

3.3.2 Accuracy

Accuracy is determined and reported as the percent recovery from the analysis of reference material, matrix spikes (MS), matrix spike duplicates (MSD), and/or laboratory control samples (LCSs). Data with acceptable quality shall meet the accuracy criteria established in this QAPP.

3.3.3 Completeness

Completeness is determined for three separate but integrated functions.

- Sample Collection completeness is calculated by comparing the number of samples
 actually collected in the field to the number of samples planned to be collected by the
 site-specific SAP. Acceptance criteria for sample collection completeness shall be
 95%.
- Acceptable Data Completeness is defined as the percentage of useable data versus the total amount of data generated. Acceptable data are generated following a review of the data using the analytical method criteria. The data generated for this project are based upon SW-846 methodology, which will be used as the method criteria. Acceptable data are all data that have completed the review or validation process and have not been rejected. Acceptance criteria for acceptable data completeness shall be 95% for each analytical method mentioned in the site-specific SAP.
- Quality Data Completeness is defined as the percentage of quality data versus the
 total set of data. Quality data are analytical data obtain from a sample delivery group
 which meet all batch quality control criteria. Completeness criteria for quality data
 shall be 80%.

3.3.4 Representativeness and Comparability

Representativeness and Comparability are both qualitative statements about the data quality. These parameters can be met if the sampling set is adequately prepared and standard methods of analysis are used for chemical analysis.

3.3.5 Sensitivity

Sensitivity is a quantitative reflection of the method detection limit (MDL) calculated by the performing analytical laboratory in accordance with 40 CRF Part 136 Appendix B.

4.0 Sampling Locations and Procedures

The investigations to be performed at OU-1 site shall produce groundwater, surface water, sediment samples, and investigation-derived waste (IDW) samples for analyses. Additional portions of select samples will be collected to complete field QC duplicate, field blank, and QA split sample analytical requirements. The specific numbers of samples (including parameters and methods) planned are provided in the FSP portion of this SAP. Investigation samples shall require analysis for as represented on Tables 3-1 and 3-2. Sampling procedures for the various media under investigation are discussed in Chapter 4.0 of the FSP.

The primary field equipment and the supporting materials necessary for the field activities are presented within Chapter 4.0 of the FSP portion of this SAP. Several different types of field measurements will be performed during these investigations. A description of the field instruments and associated calibration requirements and performance checks to be used for field measurements is presented in Chapter 12.0 of the FSP.

The locations of the sampling stations and sample media to be collected during these investigations, and the rationales for the selection of these stations, are presented in Chapter 4.0 of the FSP.

4.1 General Information and Definitions

The following subsections present general information and definitions regarding this QAPP.

4.1.1 Contractor Laboratory

Any laboratory subcontracted to perform analysis of samples will be selected through the NJDEP procurement and review process and approved by the NJDEP and USEPA prior to field mobilization.

4.1.2 Quality Assurance and Quality Control Samples

These samples are analyzed for the purpose of assessing the quality of the sampling effort and of the reported analytical data. QA and QC samples to be used for this project are field duplicates, equipment rinsate blanks, trip blanks, field blanks, and split samples. These QA/QC samples may be collected at a ratio of 1:10 per field sample, at the discretion of NJDEP. In other words,

for every 10 field samples collected, one field duplicate, one equipment rinsate, and one QA split sample may also be collected at the discretion of NJDEP.

4.1.3 Field Duplicate Quality Control Samples

The sampling team shall collect field duplicates for analysis by the on-site laboratory or contract laboratory. The purpose of these samples is to provide site-specific, field-originated information regarding the homogeneity of the sampled matrix and the precision of the field sampling effort. These samples are collected concurrently with the primary environmental samples and will equally represent the medium at a given time and location. Duplicate samples will be collected from each media addressed by this project and be submitted to the contractor laboratory for analysis. The identity of duplicate QC samples is blind to the analysts.

4.1.4 Quality Assurance Split Samples

Split samples may be collected by the sampling team and sent to an approved QA laboratory for analysis to provide an independent assessment of the contractor's and contractor laboratory's performance at the discretion of NJDEP. The contractor will coordinate with the designated QA laboratory not less than 48 hours before sampling to ensure that the laboratory is alerted to receive the QA samples and process them within the time limits specified by applicable regulations and guidelines.

4.1.5 Equipment Rinsate Blanks

These samples will be taken from the water rinsate collected during equipment decontamination activities. Rinsate blanks will consist of samples of analyte-free water, which have been rinsed over decontaminated sampling equipment, collected, and subsequently submitted for analysis of the parameters of interest. Equipment rinsates are employed to assess the effectiveness of the decontamination process, the potential for cross contamination between sampling locations and incidental field contamination.

4.1.6 Field Blanks

A sample from the site water supply used for equipment decontamination, well development, and other activities will be acquired and submitted for analysis with the primary samples. In addition, samples of on-site analyte-free water sources may also be submitted for analysis.

4.2 Sample Containers, Preservation Procedures, and Holding Times

Sample containers, chemical preservation techniques, and holding times for soils and waters samples collected during these investigations are described on Tables 4-1 and 4-2. The specific number of containers required for this study will be estimated and supplied by the analytical facilities. All bottles will be prepared I accordance with the OSWER Directive 9240,0-05A "Specifications and Guidance for Contaminant-Free Sample Containers." Additional sample volumes will be collected and provided, when necessary, for the express purpose of performing associated laboratory QC (laboratory duplicates, MS/MSD).

All sample containers will be provided by the analytical support laboratory. They will also provide the required types and volumes of preservatives with containers when they are delivered to the contractor. Temperature preservation will be maintained at 4°C (±2°C) immediately after collection and will be maintained within this temperature range until the samples are analyzed. In the event that sample integrity, such as holding times, cooler temperatures, etc., is compromised, re-sampling will occur as directed by the NJDEP/USEPA Project Manager or Project Chemist. Any affected data will be flagged and qualified per data review / validation instructions and guidance as provided in *CENWK-EC-EF Data Quality Evaluation Guidance*, dated July 26, 1999 located in Appendix B.

5.0 Sample Custody and Holding Times

It is the intent of this investigation to follow EPA policy regarding sample custody and COC protocols as described in *NEIC Policies and Procedures* (EPA 1985). Accordingly, protocols are presented for three stages of sample custody: sample collection, laboratory analysis, and final evidence files.

A sample or evidence file is under your custody when it is:

- In your possession;
- In your view, after being in your possession;
- In your possession and you place them in a secured location; or
- In a designated secure area.

5.1 Sample Collection Documentation

The sample collection documentation, sample packaging, and shipment procedures summarized below, and detailed in Chapters 5.0 and 6.0 of the FSP, will ensure that samples will arrive at the laboratory with the COC intact. The protocols for specific sample identifiers and other sample designations will be provided in a separate SOP, if necessary.

5.1.1 Field Procedures

The field supervisor is responsible for the care and custody of the samples until they are transferred or properly dispatched to the overnight carrier. As few people as possible should handle the samples. Each sample container will be labeled with a sample number, date and time of collection, sampler, and sampling location. Sample labels are to be completed for each sample. The Contractor Project Manager, in conjunction with the NJDEP/USEPA, will review all field activities to determine whether proper custody procedures were followed during the field work process and to decide if additional samples are required.

5.1.2 Field Activity Daily Logs/Documentation

Samples shall be collected following the sampling procedures documented in Chapter 4.0 of this FSP. When a sample is collected or a measurement is made, a detailed description of the location shall be recorded on a Field Activity Daily Log (FADL) or bound logbook. An example of which may be found as Figure 5-2 in the FSP. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was

collected, volume, and number of containers. A sample identification number will be assigned before sample collection. Field duplicate samples and QA split samples, which will receive entirely separate sample identification numbers, will be noted under sample description. Equipment employed to make field measurements will be identified along with their calibration dates.

5.1.3 Transfer of Custody and Shipment Procedures

A properly completed COC form shall accompany all samples collected for chemical analysis. The sample numbers and locations will be listed on the COC form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record will document transfer of custody of samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area. An example of the two-page COC form used for this investigation is presented as Figures 5-1a and 5-1b of the FSP. Additional information regarding COC practices is presented in Chapter 5.0 of the FSP.

The COC form identifying the contents will accompany all shipments. The original record will accompany the shipment, and the sample team will retain copies. The copies will be returned to project management office for the project file. Whenever co-located or split samples are collected for comparison analysis by the NJDEP/USEPA QA Laboratory or another government agency, a separate COC will be prepared for those samples and marked to indicate with whom the samples are being split.

All shipments will be in compliance with applicable U.S. Department of Transportation (DOT) regulations for environmental samples. The contractor will discourage the shipping of samples on Fridays unless it is absolutely necessary and the laboratory has assured the contractor that personnel will be present on Saturdays to receive and effect any necessary processing within the analytical holding times.

5.2 Laboratory Chain-Of-Custody Procedures

Laboratory custody procedures for analytical samples are described in Section 7.0 of the laboratory QAP, as supplied in Appendix A. These documents will identify the laboratory custody procedures for sample receipt and log-in, sample storage, tracking during sample preparation and analysis, and laboratory storage of data.

5.2.1 Cooler Receipt Checklist

The condition of shipping coolers and enclosed sample containers will be documented upon receipt at the analytical laboratory. This documentation will be accomplished using the cooler receipt checklist presented as Figure 5-2. A supply of these checklists will be provided to the subcontracted laboratory at the start of the project. A copy of the checklist will be faxed to the project chemist's office, immediately after being completed by the laboratory. The original completed checklist will be transmitted with the final analytical results from the laboratory.

5.2.2 Laboratory Internal Chain of Custody

It is expected that the subcontracted laboratory will maintain an internal chain of custody to track the location and possession of all samples at all times during the analytical process. The internal chain shall be initiated by the sample management team and continue with the request by the preparation or analytical section and shall follow the sample throughout its lifetime in the laboratory. The internal COC shall be an integral portion of the final analytical data package. Signatures from and to sample receiving should always be the beginning and end of an internal COC. Sample container or sample disposal must be documented on a COC-like form.

5.2.3 Letter of Receipt

The subcontracted laboratory shall confirm sample receipt and log-in information through transmission of a Letter-of-Receipt (LOR) to the contractor's Project Chemist. This will include returning a copy of the completed COC, a copy of the cooler receipt checklist, and confirmation of the analytical log-in indicating laboratory sample and sample delivery group numbers.

5.3 Final Evidence Files Custody Procedures

Final evidence files, including originals of laboratory reports and electronic files, are maintained under document control in a secure area. The contractor is the custodian of the evidence files and will maintain the contents of evidence files for these investigations, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports, correspondence, laboratory logbooks, and COC forms. The evidence files will be stored in a secure, limited-access area and under custody of the contractor's Project Manager.

Analytical laboratories will retain all original raw data information (both hard copy and electronic) in a secure, limited-access area and under custody of the Laboratory Project Manager.

6.0 Analytical Procedures

All samples collected during the investigation activities will be analyzed by laboratories reviewed and validated by NJDEP or equivalent. QA samples shall be collected from groundwater, surface water, and sediment sample locations and analyzed by the designated QA Laboratory. Each laboratory supporting this work shall provide statements of qualifications including organizational structure, QA Manual, and standard operating procedures (SOPs).

6.1 Field Screening Analytical Procedures

Procedures for field measurement are described in Chapter 4.0 of the FSP. A listing of the methodologies appears on Tables 3-1 and 3-2.

6.2 Subcontract Analytical Procedures

Samples collected during the project will be analyzed by EPA methods and other nationally recognized methods. EMSL SOPs are based on the methods published by the USEPA in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW846*, Third/Fourth Edition (November 1986; Revision 1, July 1992; Revision 2, November 1992; and Updates 1, 2, and 3) and are incorporated into this document as a reference. The reporting limits (RL) are project specific, but may vary due to laboratory performance during their MDL studies. The actual RL is 3 to 5 times the calculated MDL.

The subcontracted laboratory shall not subcontract or transfer any portion of this work to another facility, unless expressly permitted to do so in writing by the contractor and the NJDEP/USEPA Project Manager.

If contaminant concentrations are high, or matrices other than normal waters and soils are required for analysis, the analytical protocols may be inadequate. In these cases, sample analysis may require modifications to defined methodology. Any proposed changes to analytical methods specified require written approval from the contractor and the NJDEP/USEPA. All analytical method variations will be identified in investigation-specific addenda. These may be submitted for regulatory review and approval when directed by the contractor Project Manager.

These SOPs must be adapted from and reference standard EPA SW-846 methods and thereby specify:

- Procedures for sample preparation
- Instrument start-up and performance check
- Procedures to establish the actual and required detection limits for each parameter
- Initial and continuing calibration check requirements
- Specific methods for each sample matrix type
- Required analyses and QC requirements.

6.2.1 Preparation Procedures

Extraction and digestion procedures for the preparation of liquid and solid matrices discussed in this section are presented in Table 6-1. The appropriate method will be supplied in the laboratory- specific SOP.

6.2.2 Analytical Procedures

Brief descriptions for each analytical method are included in the following subsections

6.2.2.1 Method 100.2 - Asbestos Compounds in Groundwater

Method 100.2 is used to determine part per billion (ppb) level concentrations of certain asbestos containing residues in water samples. Prior to using the method, appropriate sample preparation techniques must be applied and adhered to during execution.

The aqueous sample is to be preserved on ice at 4 degrees C and filtered within 48 hours by the laboratory. The aqueous sample is filtered through a 0.1 micro millimeter capillary pore polycarbonate filter after which the filter is prepared by carbon extraction replication for examination in a transmission electron microscope (TEM). Fibers are classified using selected area electron diffraction (SAED) and energy dispersive X-ray analysis (EDXA). Measurement of characteristic features on a recorded and calibrated SAED pattern is specified for precise composition, and quantitative interpretation of at least one calibrated zone axis SAED pattern are specified for precise identification of amphibole (i.e. amosite, crocidolite, actinolite, tremolite, and anthophylilite). Amphibole identification procedures and generation of the standard reporting format specified for the fiber count results are achieved using two computer programs that are integral to the analytical method.

6.2.2.2 Method 600/R-93-116 Asbestos in Bulk Building Materials

This method describes the procedures for the determination of the presence or absence of asbestos in bulk samples of building material. Samples are initially examined under low magnification using a stereo microscope, contained in a hood equipped with a HEPA filter. Initial observations should note gross material appearance (homogeneity, fibrous/non-fibrous)

and physical characteristics (color, texture, friable/non-friable).

Analysis by polarized light microscopy (PLM) is used for the positive identification of suspect fibers. Positive identification of asbestos requires the determination of several optical properties peculiar to the six types of asbestos: chrysotile asbestos, grunerite asbestos (amosite), riebeckite asbestos (crocidolite), anthophyllite asbestos, tremolite asbestos and actinolite asbestos.

Quantitative estimates of the asbestos content, and other major constituents, of the sample are made based on a combination of the estimates from both the gross and the PLM examinations.

Interference from other inorganic and organic fibrous constituents, cleavage fragments of natural minerals, binders, coatings, and man-made fibers may be encountered. Moisture may interfere with the determination of some optical properties. Therefore, wet samples should be dried prior to analysis.

The sample matrix may cause a variety of interferences under PLM observation. Special matrix reduction techniques may be necessary to reduce these interferences.

7.0 Calibration Procedures and Frequency

This chapter describes procedures for maintaining the accuracy of all the instruments and measuring equipment used for conducting laboratory analyses. These instruments and equipment shall be calibrated before each use or on a scheduled, periodic basis according to manufacturer instructions. Specific laboratory details are provided in Section 9.0 of the EMSL QAP, found in Appendix A.

7.1 Analytical Support Areas

The following sections discuss the calibration needs for operations within the analytical laboratory necessary to support the instrumentation portion.

7.1.1 Analytical Standards

All primary reference and secondary working standards used for the purpose of instrument calibration and recovery determinations must be to be traceable to National Institute of Standards and Technology (NIST) or USEPA sources. The preparation and use of these standards must be documented in a standards logbook which shall include the preparer's name, date of preparation, and date of expiration and storage location.

7.1.2 Laboratory Balances

All balances to be used for sample weights and/or standards preparation must receive an annual manufacturer's calibration. The balance must be calibrated daily with a minimum of two (preferably three) Class "S" weights which bracket the range of weights to be determined. A hardbound balance logbook must be maintained with the results of the daily calibrations.

7.1.3 Laboratory Refrigerators/Freezers

All cold storage units (for both samples and standards) must be monitored daily for proper use. The acceptable working range of the unit must be clearly posted on the unit's front panel. All thermometers used for monitoring must be immersion type and be calibrated against a certified thermometer on a yearly basis.

7.1.4 Laboratory Water Supply

The laboratory water unit shall be capable of supplying sufficient quantities of American Society for Testing and Materials (ASTM) Type II reagent water (resistivity of >1 megohm-cm @25°C)

to the required analytical areas. Recommendations for "polishing" water for analytical use are ion-exchange units for inorganic analyses and distillation/deionization followed by UV treatment or carbon absorption for organic analyses. Conductivity or resistance reading of the system water shall be documented daily, at a minimum or greater dependant upon the water usage.

7.2 Laboratory Analytical Instrumentation

Calibration of laboratory equipment will be based on approved written SOPs. Records of calibration, repairs, or replacement will be filed and maintained by laboratory personnel performing QC activities. These records will be filed at the location where the work is performed and will be subject to QA audit. Procedures and records of calibration will follow NJDEP/USEPA and the contractor-reviewed OAP.

In all cases where analyses are conducted according to SW846 protocols, the calibration procedures and frequencies specified in the methods will be followed exactly. For analyses governed by SOPs, refer to the appropriate SOP for the required calibration procedures and frequencies.

Records of calibration will be kept as follows:

- Each instrument will have a record of calibration with an assigned record number.
- A label will be affixed to each instrument showing identification numbers, manufacturer, model numbers, date of last calibration, signature of calibrating analyst, and due date of next calibration. Reports and compensation or correction figures will be maintained with the instrument.
- A written step-wise calibration procedure will be available for each piece of test and measurement equipment.
- Any instrument that is not calibrated to the manufacturer's original specification will display a warning tag to alert the analyst that the device carries only a "Limited Calibration."

Details of EMSL's calibration procedures and frequency are provided in Section 21.0 of their QAP. Actual details of the calibration requirements shall be found in the laboratory-specific SOPs presented in Appendix C.

7.2.1 High Performance Liquid Chromatography (HPLC)

Initial calibration consists of a minimum five-point standard curve of all target compounds within the linear range of the specific detector. The lowest standard in the calibration shall be no greater than 2-times the reporting limit (preferably at or below the reporting limit). The acceptability of the curve is based on either the percent Relative Standard Deviation (%RSD) of the response for each compound calibrated, or the linear regression of the data points for each compound. Following the initial calibration of the instrument, linearity of the curve shall be checked with a second source calibration standard. The curve and instrument response shall be checked during the analytical sequence (preferably after every 10 samples). Acceptability of the continuing calibration check is based upon either percent recovery or percent difference.

7.2.2 Inductively Coupled Plasma Optical Emission Spectroscopy (ICP or ICP-OES)

The ICP instrument shall be calibrated with a minimum of 3 standards plus a blank. Calculation of the curve is determined by linear regression using a correlation coefficient (r) ≥ 0.995 as acceptance criteria. Following the calibration, the highest standard must be rerun and agree within 5% of the true concentration. The instrument calibration shall be verified with an Initial Calibration Verification (ICV) standard at the midpoint of the range of the curve. Agreement of the ICV must be within ±10% of the true concentration. Following the ICV, the Initial Calibration Blank (ICB) is run to provide instrument response with no analyte present. The Interference Check Standards (ICS-A and ICS-AB) are then run to first determine the recovery of the major cations and then the recovery of the minor cations in the presence of the major cations. Acceptable recovery of the ICS is within 80-120% of the true values for both the major and minor cations. To provide information regarding the low end of the calibration curve response, a low-level standard is run. Presently, there are no acceptance criteria for this determination, but is used to provide information about recovery at the low end. After every 10 samples, a Continuing Calibration Verification (CCV) standard and Continuing Calibration Blank (CCB) are run to determine curve acceptability. This is continued until all samples are analyzed. Recalibration of the instrument and reanalysis of samples shall be performed if the ICS-A, ICS-AB, or CCV fails criteria.

7.2.3 Gas Chromatography / Mass Spectrometry (GC / MS)

The GC/MS instrument shall be initially tuned with decafluorotriphenylphosphine, DFTPP, and meet certain acceptance criteria. Following successful tuning of the instrument, it is calibrated with a minimum of 5 standards. Calculation of the linearity of the curve is determined either by

Mean Relative Response Factor (RRF) or linear regression using a correlation coefficient (r) ≥0.995 as acceptance criteria. Mean RRF calculation is acceptable if the relative standard deviation (%RSD) average of all the RRFs is less than 15%. Following the successful calibration, samples are run until a twelve-hour period has expired. After the twelve-hour period the instrument must again be successfully tuned and followed with a continuing calibration standard (mid-point calibration standard).

8.0 Internal Quality Control Checks

Internal quality control checks are generated by the analytical laboratory and are used to determine whether an analytical operation is in control or if the sample matrix has an effect on the data being generated. Internal QC provides data quality consistent with the intended purpose of the sample collection. The NJDEP/USACE QA Laboratory shall provide external QA. The external QA laboratory shall receive the identified QA sample splits.

8.1 Field Sample Collection

Collecting field duplicates and trip blanks in accordance with the procedures described in the project FSP accomplishes the assessment of field sampling precision and accuracy.

NJDEP/USACE protocol requires the collection of field QA/QC at the specified rate per sampling event. Therefore, for every sampling event, a field duplicate, field blank, and rinsate or equipment blank shall be collected to determine assess the impact of field conditions upon the analytical data.

8.2 Laboratory Analysis

Analytical QC procedures for the OU-1 investigations are specified in the analytical tables presented in Chapter 6.0 of this QAPP and within the individual method descriptions. These specifications include the types of QC checks normally required: method blanks calibration standards, calibration check standards, and laboratory duplicate analysis. Calibration compounds and concentrations to be used and the method of QC acceptance criteria for these parameters have been identified.

To ensure the production of analytical data of known and documented quality, laboratories associated with these investigations will implement all method QA and QC checks.

The referenced analytical laboratory has provided a written QAP (Appendix C) that provides rules and guidelines to ensure the reliability and validity of work conducted at the laboratory. Compliance with the QAP is coordinated and monitored by the laboratory's QA department, which is independent of the operating departments. For this investigation, the qualified contract analytical laboratory's QAP will be referenced and implemented in its entirety.

EMSL's QAP provides objectives to:

- provide a controlled, traceable link through the entire determination process (sample collection through reporting)
- provide a predetermined program for the acceptance or rejection of analytical data
- provide a system in which the laboratory can take and document action necessary to correct problems and insure the validity of reported laboratory data
- estimate the level of quality of each analytical system in a timely, efficient, and costeffective manner
- provide a system which is able to assist in early recognition of deficiencies which might affect the quality of data
- have in place a quality assurance audit program to insure that the plan, as established, is implemented and needed updates are made.

All laboratory procedures are documented in writing as SOPs, which are edited and controlled by the QA department. The specific laboratory analytical SOPs are presented in Appendix C. Internal QC measures for analysis will be conducted with their SOPs and the individual method requirements specified.

8.3 Internal Quality Control Checks

Implementation of QC procedures during sample collection, analysis, and reporting ensures that the data obtained are consistent with its intended use. Both field QC and laboratory QC checks are performed throughout the work effort to generate data confidence. Analytical QC measures are used to determine if the analytical process is in control, as well as to determine the sample matrix effects on the data being generated.

Specifications include the types of QC required (duplicates, sample spikes, surrogate spikes, reference samples, controls, blanks, etc.), the frequency for implementation of each QC measure, compounds to be used for sample spikes and surrogate spikes, and the acceptance criteria for this QC.

The laboratory shall provide documentation in each data package that both initial and ongoing instrument and analytical QC functions have been met. The laboratory will reanalyze any non-conforming analysis, if sufficient sample volume is available. It is expected that sufficient sample volumes will be collected to provide for reanalysis, if required.

8.3.1 Batch Quality Control

Sample batch QC can either be associated with sample preparation or with the analytical determination. In either case the batch is not to exceed twenty samples of similar matrix. The preparation batch is the set of samples that are extracted or digested together by the same laboratory technician, with the same lot of reagents, and during the same time period. All the samples within the same preparation batch must be of the same matrix and must have its own unique method blank and QC samples as defined in the following sections. The analytical batch is the group of samples that are analyzed together during the same analytical sequence within one continuous time period. The analytical batch can consist of multiple preparation batches, but must analyze all constituents of the preparation batch. QC cannot be run separately from the analytical samples.

8.3.1.1 Method Blanks

One type of method blank is the preparation (prep) blank. The prep blank is a sample of a pure non-contaminated matrix of interest (usually reagent grade water or purified silica sand) that is subjected to all of the sample preparation (digestion, distillation, extraction) and analytical methodology applied to the samples. The preparation blank is used to assess the level of background contamination that might affect low-level concentration results. The affect to low concentration samples could be:

- false positive results, i.e., reported detects for non-detect parameters
- biased high low concentration results, i.e., higher detected quantities than really present.

This type of method blank must be prepared and analyzed with each analytical sample batch.

The second type of method blank is the instrument blank, which is either an aliquot of neat reagents or reagent water that is analyzed prior to samples to establish background levels of the analytical system.

Analytical sensitivity goals are identified in Chapter 6.0 tables as reporting limits (practical quantitation limits). Method blank levels should be below these levels for all analytes. Contamination levels reported in the blanks are **never** subtracted from the sample's reported concentration.

8.3.2 Matrix-Specific Quality Control

Matrix-specific QC is based upon precision and accuracy performance of actual environmental samples. Sample duplicates are examples of matrix-specific QC.

8.3.2.1 Laboratory Duplicates

Laboratory duplicates are separate aliquots of a single sample that are prepared and analyzed concurrently by the laboratory. This duplicate sample shall not be a method blank, trip blank, or field blank. The primary purpose of the laboratory duplicate is to check the precision of the laboratory analyst, the sample preparation methodology, and the analytical methodology. If there are significant differences between the duplicates, the affected analytical results will be re-examined. One sample per 10 samples will be a laboratory duplicate, with fractions rounded to the next whole number.

8.3.2.4 Method-Specific Quality Control

The laboratory must follow specific quality processes as defined by the method.

9.0 Calculation of Data Quality Indicators

The following sections present the calculation of data quality indicators.

9.1 Field Measurements Data

Field data will be assessed by the Project Chemist or designee. The review will assess the field results for compliance with the established QC criteria that are specified in the QAPP and FSP. Accuracy of the field measurements will be assessed using daily instrument calibration, calibration check, and analysis of blanks. Precision will be assessed on the basis of reproducibility by multiple reading of a single sample.

Field data completeness will be calculated using the Equations below.

Sample Collection (1a):

% Completeness =
$$\frac{\text{Number of Sample Points Collected}}{\text{Number of Sample Points Planned}} \times 100$$

Field Measurements (1b):

% Completeness =
$$\frac{\text{Number of Valid Field Measurements Made}}{\text{Number of Field Measurements Planned}} \times 100$$

9.2 Laboratory Data

Laboratory results will be assessed for compliance with required precision, accuracy, representativeness, completeness, comparability and sensitivity as follows. Additional details can be found in EMSL's QAP.

9.2.1 Precision

The precision of the laboratory analytical process will be determined through evaluation of the sample and sample duplicate analyses.

Investigative sample matrix precision will be assessed by comparing the analytical results between laboratory duplicate analyses for inorganic analysis. The RPD will be calculated for each pair of duplicate analysis using the appropriate formula in Table 9-1 and produce an

absolute value for RPD. This precision measurement will include variables associated with the analytical process and sample heterogeneity.

9.2.2 Accuracy

The accuracy of the laboratory analytical measurement process will be determined by comparing the percent recovery for the LCS / LCSD versus its documented true value. The EMSL QAP discusses this item in detail.

Overall project accuracy include the assessment of investigative sample using the analytical results of MS and MSD samples. The %R of LCS and MS/MSD samples will be calculated using the appropriate formula on Table 9-1. This overall accuracy will include variables associated with the analytical process, influences related to sample matrix interferences, and sample heterogeneity. It is theorized that the lead recovery for the stabilized soil material may be compromised due the presence of excess sulfate in the stabilizing component. Pre-project testing is planned to test this theory. Therefore, if proven, the recovery of the laboratory control samples will be the main source of accuracy measurements.

9.2.3 Completeness

Data completeness of laboratory analyses will be assessed for compliance with the amount of data required for decision making. The completeness is calculated using the following equation:

% Completeness =
$$\frac{\text{Number of Valid Results}}{\text{Number of Possible Results}} \times 100$$

Completeness criteria have been applied to several activities during the remedial action. See Chapter 3.3 for an extended discussion on the various completeness calculations.

9.2.4 Sensitivity

Sensitivity of the analytical determination is directly reported to the laboratory's MDL. Achieving MDL depends on sample preparation techniques, instrumental sensitivity, and matrix effects. Therefore, it is important to determine actual MDLs through the procedures outlined in 40 CFR 136, Appendix B. MDLs should be established for each major matrix under investigation (i.e., water, soil) through multiple determinations, leading to a statistical evaluation of the MDL.

It is important to monitor instrument sensitivity through calibration blanks and low concentration standards to ensure consistent instrument performance. It is also critical to monitor the analytical method sensitivity through analysis of method blanks, calibration check samples, and LCSs, etc.

9.3 Project Completeness

Project completeness will be determined by evaluating the planned versus actual data. Consideration will be given for project changes and alterations during implementation. All data not flagged as rejected (R-qualified) by the review, verification, validation, or assessment processes will be considered valid. Overall, the project completeness will be assessed relative to media, analyte, and area of investigation. Completeness objectives are listed on Table 3-2 (water).

9.4 Representativeness/Comparability

Representativeness expresses the degree to which data accurately reflect the analyte or parameter of interest for the environmental media examined at the site. It is a qualitative term most concerned with the proper design of the sampling program. Factors affecting the representativeness of analytical data include appropriate sample population definitions, proper sample collection and preservation techniques, analytical holding times, use of standard analytical methods, and determination of matrix or analyte interferences. Sample collection, preservation, analytical holding time, analytical method application, and matrix interferences will be evaluated by reviewing project documentation and QC analyses. The EMSL QAP will provide details on these items.

Comparability, like representativeness, is a qualitative term relative to the confidence of how one project data set compares with another. The comparability issue is controlled through the use of defined sampling methodologies, use of standard sampling devices, standard analytical protocols/procedures, and QC checks with standard control limits. Through proper implementation and documentation of these standard practices, the project will establish confidence that data will be comparable to other project and programmatic information.

Additional input to determine representativeness and comparability may be gained through statistical evaluation of data populations, chemical charge balances, compound evaluations, or dual measurement comparisons.

10.0 Corrective Actions

Corrective actions may be required for two major types of problems: analytical/equipment problems and noncompliance with criteria. Analytical and equipment problems may occur during sampling, sample handling, sample preparation, laboratory instrumental analysis, and data review. Discussion of the analytical laboratory's corrective actions can be found in EMSL's QAP (Appendix C).

10.1 General Field Issues

All nonconformance situations noted during the sampling phase of the project operation shall be documented and acted upon through a formal corrective action program. The person identifying the problem is responsible for notifying the Field Supervisor and ultimately the contractor Project Manager and the NJDEP/USEPA Project Manager. When the problem is analytical in nature, information on these problems will be promptly communicated to the contractor Project Chemist. Implementation of corrective action will be confirmed in writing by the laboratory QAM to the contractor-Project Chemist.

Any nonconformance issue in conflict with the established QC procedures in the SAP will be identified and corrected in accordance with this section of the QAPP. The contractor Project Manager or their designee will issue a non-conformance report (NCR) for each nonconforming condition. Figure 10-1 of the FSP presents an NCR.

Corrective actions will be implemented and documented on a FADL or in a field logbook. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are deemed insufficient, work may be stopped through a stop-work order issued by the contractor Project Manager and the NJDEP/USEPA Project Manager.

For unexpected situations encountered during field activities whereby changes to operating system are necessary to implement, a Field Work Variance (FWV) will be issued. All variances from existing operating procedures, field sampling plan, quality assurance requirements, and/or health and safety plans will be documented on a FWV (Figure 10-2 of the FSP).

10.2 Laboratory Analyses

Each site-specific investigation laboratory QA plan shall provide systematic procedures to identify laboratory related out-of-control situations and corrective actions. Corrective actions shall be implemented to resolve problems and restore malfunctioning analytical systems. Laboratory personnel shall have received QA training and shall be aware that corrective actions are necessary when:

- QC data are outside warning or control windows for precision and accuracy
- Blanks contain target analytes above acceptable levels and must be investigated
- Undesirable trends are detected in spike recoveries or RPD between duplicates
- There are unusual changes in detection limits
- Deficiencies are detected by internal audits, external audits, or from performance evaluation samples results
- Inquiries concerning data quality are received.

Corrective action procedures are often handled at the bench level by the analyst who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the Laboratory Director, and the QAM for further investigation. Once resolved, full documentation of the corrective action procedure is filed with project records and the QA Department, and the information is summarized within case narratives.

10.2.1 Incoming Samples

Problems noted during sample receipt will be documented in the appropriate laboratory LOR. The contractor and NJDEP/USEPA chemists and project managers will be contacted immediately to determine the resolution. All corrective actions will be thoroughly documented.

10.2.2 Sample Holding Times

When sample extraction/digestion or analytical analysis is not performed within method required holding time specifications, the contractor and NJDEP/USEPA Project Chemists will be notified immediately to determine the resolution. All corrective actions will be thoroughly documented.

10.2.3 Instrument Calibration

Instrumentation that fails to meet standardization or calibration criteria shall not analyze project samples. All project samples will be reanalyzed if initially performed following an initial and/or continuing calibration analytical sequence that does not meet method requirements. Corrective action may require standard re-preparation, instrument maintenance, and instrument recalibration/restandardization.

10.2.4 Practical Quantitation/Reporting Limits

All appropriate measures shall be required to prepare and clean up samples in an attempt to achieve the practical quantitation / reporting limits. When difficulties arise in achieving these limits, the laboratory will notify the contractor and NJDEP/USEPA project chemists to determine the resolution. All corrective actions shall be thoroughly documented.

Any dilutions impacting the practical quantitation limits will be documented in case narratives along with revised quantitation limits for those analytes affected. Analytes detected above the method detection limits, but below the practical quantitation limits, will be reported as estimated values. Both the undiluted and diluted set of data shall be provided to the contractor.

10.2.5 Method Quality Control

Failure of method-required QC to meet the requirements specified in this project QAPP shall require corrective actions for all affected data. Resulting corrective actions may include those listed in Section 10.2.6. The contractor and NJDEP/USEPA project chemists will be notified as soon as possible to discuss possible corrective actions, particularly when unusual or difficult sample matrices are encountered.

10.2.6 Calculation Errors

When calculation or reporting errors are noted within any given data package, reports will be reissued with applicable corrections. Case narratives will clearly state the reasons for re-issuance of reports.

Corrective actions may include:

- re-analyzing the samples, if holding time criteria permit
- evaluating blank contaminant sources, elimination of these sources, and reanalysis
- modifying the analytical method (i.e., standard additions) with appropriate notification and documentation
- re-sampling and analyzing

- evaluating and amending sampling procedures
- accepting data and acknowledging the level of uncertainty.

If re-sampling is deemed necessary due to laboratory problems, the contractor Project Manager will identify the necessary cost recovery approach to implement the additional sampling effort.

11.0 Data Reduction, Validation, and Reporting

This chapter describes the data review process enacted to ensure validity and usability of the subcontracted analytical data. Prior to its submittal to the contractor, the laboratory technical personnel will initially review all data generated by the laboratory. This review will provide a check to ensure the correctness of the reported results and generate a case narrative to explain any anomalies that may affect the validity or usability of the data. Following receipt of the data package, the electronic data will be validated by the database and the hardcopy data will be reviewed or validated by the contractor's chemists or designees.

11.1 Data Reduction

All raw data generated from the OU-1 project will be reduced by the laboratory to provide a documented CLP-like data package to the contractor and NJDEP/USEPA project management team.

11.1.1 Field Measurements and Sample Collection

Raw data from field measurements and sample collection activities will be appropriately recorded in field logbooks or FADLs. Data to be used in project reports will be reduced and summarized. The methods of data reduction will be documented.

The contractor Project Manager or designee is responsible for data review of all field-generated data. This includes verifying that all field descriptive data are recorded properly, that all field instrument calibration requirements have been met, that all field QC data have met frequency and criteria goals, and that field data are entered accurately in all logbooks and worksheets.

11.1.2 Laboratory Services

All samples collected for these investigations will be sent to an approved subcontracted laboratory. Data reduction, evaluation, and reporting of samples analyzed by the laboratory will be performed according to specifications outlined in both Section 22.0 of the laboratory's QA plan and this QAPP. Laboratory reports shall include documentation verifying analytical holding time compliance, method blank results, summarized QA/QC results, raw data, preparation logs and analytical run-logs.

Laboratories will perform in-house analytical data reduction under the direction of their Laboratory QA Manager. The Laboratory QA Manager or designee is ultimately responsible for

assessing data quality and informing the contractor and NJDEP/USEPA of any data that are considered unacceptable or require caution on the part of the data user in terms of its reliability. Data will be reduced, reviewed, and reported as described in the laboratory QA plan. Data reduction, review, and reporting activities performed by the laboratory are summarized below and detailed in EMSL's QAP:

- The analyst who generated the raw data has the primary responsibility for the accuracy and completeness of the data. All data will be generated and reduced following the QAPP defined methods and implementing laboratory SOP protocols.
- A peer analyst performs a Level 1 technical data review consistent with an established set of guidelines. The review shall ensure the completeness and correctness of the data while assuring all method QC measures have been implemented and were within appropriate criteria. Items to be reviewed include: preparation logs, analysis runs, methodology, results, quality control results, internal QC checks, checklists and signoff sheets.
- The area supervisor or data review specialist will complete the Level 2 technical review. This level reviews the data for attainment of QC criteria as outlined in the established methods and for overall reasonableness. It will ensure all calibration and QC data are in compliance, qualitative identification of compounds is correct, quantitative calculations are correct, and check at least 10 percent of the data calculations. This review shall document that the data package is complete and ready for reporting and archival.
- Upon acceptance of the raw data by the area supervisor, the report is generated and sent
 to the Laboratory Project Manager or QA representative for Level 3 administrative data
 review. This total overview review will ensure consistency and compliance with all
 laboratory instructions, the laboratory QA plan, the project laboratory SOW, and the
 project QAPP.
- The Laboratory Project Manager will complete a thorough review of all reports.
- Final reports will be generated and signed by the Laboratory Project Manager.
- Data Packages (in CLP-like style) will then be delivered to IT for data review, validation, or assessment.

The data review process will include identification of any out-of-control data points and data omissions, as well as interactions with the laboratory to correct data deficiencies. Decisions to repeat sample collection and analyses may be made by the contractor's Project Manager based on the extent of the deficiencies and their importance in the overall context of the project. The laboratory shall provide laboratory qualifiers (flags) to data that: (1) are concentrations below

required detection limit (J), (2) estimated concentration due to poor spike recovery (E/N), and (3) concentration of chemical also found in laboratory blank (B).

Laboratories will prepare and retain full analytical and QC documentation for the project. Such retained documentation will be both hard (paper) copy and electronic storage media (e.g., magnetic tape) as dictated by the analytical methodologies employed. As needed, laboratories will supply hard copies of the retained information.

Laboratories will provide the following information to the contractor in each analytical data package submitted:

- Cover sheets listing the samples included in the report and narrative comments describing problems encountered in analysis
- Tabulated results of inorganic, organic, and miscellaneous parameters identified and quantified
- Analytical results for QC sample spikes, sample duplicates, initial and continuous calibration verifications of standards and blanks, standard procedural blanks, LCSs and other deliverables as identified in Section 11.3
- Associated raw data to support the tabulated results for samples and QA/QC
- Tabulation of instrument detection limits determined in pure water.

11.2 Data Validation

Data validation is the systematic review process performed to ensure that the precision and accuracy of the analytical data are adequate for their intended use. At the present time, it was not determined how much of this data are to be validated or whether this data are to be validated. The following discussions are provided based on the premise the data are to be validated

11.2.1 Data Validation Approach

The greatest uncertainty in a measurement is often a result of the sampling process and inherent variability in the environmental media rather than the analytical measurement. Therefore, analytical data validation will be performed only to the level necessary to minimize the potential of using false positive or false negative results in the decision-making process (i.e., to ensure accurate identification of detected versus non-detected compounds). This approach is consistent

with the DQOs for the project, with the analytical methods, and for determining contaminants of concern and calculating risk.

Samples will be analyzed through use of standard analytical methods. Definitive data will be reported consistent with the deliverables identified in Section 11.3, Table 11-1. This report content is consistent with what is understood as an EPA Level IV deliverable (data forms including laboratory QC, and raw sample data including calibration information). This definitive data will then be validated through the review process presented in Section 11.2.2 and qualified using guidelines presented on Table 11-2. DQOs identified in Chapter 3.0 and method-specified criteria will be validated. The contract laboratory will retain an additional copy of the comprehensive analytical information.

11.2.2 Primary Analytical Data Validation Categories

Validation will be accomplished by comparing the contents of the data packages and QA/QC results to requirements contained in the requested analytical methods and this QAPP. The contractor validation support staff will be responsible for these activities. The protocol for analyte data validation is presented in:

- Contractor's Standard Quality Practices, Technical Procedures
- NJDEP Standard Methods for Data Validation
- SW-846 Method requirements
- EPA CLP National Functional Guidelines for Organic Data Review (EPA 1994b)
- EPA CLP National Functional Guidelines for Inorganic Data Review (EPA 1994c)
- CENWK-EC-EF-Data Quality Evaluation Guidance (USACE 1999, as a reference)

The contractor's validation support staff will conduct a systematic review of a minimum of 10% of the data for compliance. This compliance is with the established SW-846 QC criteria and not the EPA-CLP criteria. The evaluation is based on the following method-dependant categories:

- Holding times
- Blanks
- LCSs
- MS/MSD
- Surrogate recovery (organic methods)
- Internal standards (primarily organic methods)
- ICP or atomic absorption QC
- Calibration
- Chromatograms, intensity / absorbance readings, (raw data)
- Sample reanalysis

- Secondary dilutions
- Laboratory case narrative.

Consistent with the data quality requirements as defined in the DQOs, all project data and associated QC will be evaluated on these categories and qualified as per the outcome of the review. Either the contractor's or equivalent validation forms will be completed and presented with the Quality Control Summary Report (QCSR).

11.2.2.1 Holding Times

Evaluation of holding times ascertains the validity of results based on the length of time from sample collection to sample preparation or sample analysis. Verification of sample preservation must be confirmed and accounted for in the evaluation of sample holding times. The evaluation of holding times is essential to establishing sample integrity and representativeness. Concerns regarding physical, chemical, or biochemical alteration of analyte concentrations can be eliminated or qualified through this evaluation.

11.2.2.2 Blanks

The assessment of blank analyses is performed to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks applies to any blank associated with the samples, including field, equipment, and method blanks. Contamination during sampling or analysis, if not discovered, may result in false-positive data.

Blanks will be evaluated against quantitation limit goals. Analytical method blanks should be below $2\times$ these levels. Field and equipment rinsate blanks will be evaluated against $5\times$ these levels for all analytes.

11.2.2.3 Laboratory Control Samples

The LCS serves as a monitor of the overall performance of the analytical process, including sample preparation, for a given set of samples. Evaluation of this standard provides confidence in or allows for qualification of results based on a measurement of process control during each sample analysis.

11.2.2.4 Surrogate Recovery

System monitoring compounds are added to every sample, blank, matrix spike, MS, MSD, and standard prepared or analyzed for organic constituents. They are used to evaluate extraction,

cleanup, and analytical efficiency by measuring recovery on a sample-specific basis. Poor system performance as indicated by low surrogate recoveries is one of the most common reasons for data qualification. Evaluation of surrogate recovery is critical to the provision of reliable sample-specific analytical results.

11.2.2.5 Calibration

The purpose of initial and continuing calibration verification analyses is to verify the linear dynamic range and stability of instrument response. Relative instrument response is used to quantitate the analyte results. If the relative response factor is outside acceptable limits, the data quantification is uncertain and requires appropriate qualification.

11.2.2.6 Sample Reanalysis

When instrument performance-monitoring standards indicate an analysis is out of control, the laboratory is required to reanalyze the sample. Whether or not the reanalysis solves the problem (i.e., surrogate compound recoveries are outside the limits for both analyses), the laboratory is required to submit the data from all analytical runs. An independent review may be required to determine which is the appropriate sample result to report.

11.2.2.7 Secondary Dilutions

When the concentration of any analyte in any sample exceeds the initial calibration range, a new aliquot of that sample must be diluted and reanalyzed. The laboratory is required to report data from both analyses. When this occurs, an independent review of the data is required to determine the appropriate results to be used for that sample. An evaluation of each analyte exceeding the calibration range must be made, including a review of the dilution analysis performed. Results reported should be a combination of both sets of data. The original run results for the analytes within initial calibration range and the secondary dilution results for those outside the original but within the secondary.

11.2.2.8 Raw Data (inc. Chromatograms and Intensity/Absorbance Readings)

Raw data will be used to assess the qualitative and quantitative assumptions and decisions made by the laboratory and determine whether the decisions made within the laboratory can be substantiated by a third party position. Retention times and chromatographic peak shapes are verified.

11.2.2.9 Laboratory Case Narratives

Analytical laboratory case narratives are reviewed for specific information concerning the analytical process. This information is used to direct the data validator to potential problems with the data.

11.3 Data Reporting

All data generated for the OU-1 investigations will be provided in both hardcopy and electronic format. The data may be in the International Technology Environmental Management System (ITEMS) format (Table 11-3) or acceptable format. The laboratory will be required to confirm sample receipt and log-in information. The laboratory will return a copy of the completed COC and confirmation of the laboratory's analytical log-in to the contractor within 24 hours of sample receipt.

The subcontract analytical laboratory will prepare and deliver a full copy of an analytical data package similar to that required by CLP. The lab is required to retain a full copy of the analytical and QC documentation. Such retained documentation will include all hard copies and other storage media (e.g., magnetic tape). As needed, the subcontract analytical laboratory will make available all retained analytical data information.

The data shall be formatted in ITEMS format (or acceptable format) to facilitate electronic data entry, review, and evaluation. The electronic data set will be transferred automatically into the ITEMS database. Following the transfer, the data set will be validated to an equivalent EPA Level IV validation review by the validation module within ITEMS. The module will provide an error report, which includes data flags in accordance with the above-referenced protocols. The report will be accompanied with additional comments of the Data Validation Team. The associated data flags will include such items as: (1) estimated concentration below-required reporting limit; (2) estimated concentration due to poor calibration, internal standard, or surrogate recoveries; (3) estimated concentration due to poor spike recovery; and (4) presence of contaminants in the laboratory blank.

After the electronic validation has been performed, an EPA Level IV validation on a minimum of 10% of the data will be performed by qualified chemists. Flags signifying the usability of data will be noted and entered into an analytical database. Deficiencies in data deliverables will be corrected through direct communication with the field or laboratory, generating immediate response and resolution. All significant data discrepancies noted during the validation process

will be documented through NCRs, which are sent to the laboratory for clarification and correction.

Decisions to repeat sample collection and analyses may be made by the contractor's Project Manager and the Project/Program Chemist based on the extent of the deficiencies and their importance in the overall context of the project.

The contractor's data assessment will be accomplished by the joint efforts of the data validator, the Program/Project Chemist and the Project Manager. Data assessment by data management will be based on the criteria that the sample was properly collected and handled according to the FSP and Chapters 4.0 and 5.0 of this QAPP. An evaluation of data accuracy, precision, sensitivity and completeness, based on criteria in Chapter 9.0 of this QAPP, will be performed by a data assessor and presented in the QCSR. This data quality assessment will indicate that data are: (1) usable as a quantitative concentration, (2) usable with caution as an estimated concentration, or (3) unusable due to excessive out-of-control QC results.

Project investigation data sets will be available for controlled access by the contractor's Database Manager and other authorized personnel. Each data set will be incorporated into investigation reports as required.

11.4 Data Turnaround Requirements

Generally, a normal turnaround time for investigated materials for asbestos in groundwater is (14 days).

12.0 Preventive Maintenance Procedures

The following sections present the preventative maintenance procedures.

12.1 Field Instruments and Equipment

The field equipment for this project may include temperature probes; pH meters; conductivity meters; organic vapor detectors (FID or PID); screening test kits for lead; personal air sampling pumps for breathing space analysis and geophysical equipment. Specific preventive maintenance procedures to be followed for field equipment are those recommended by the manufacturers. A summary of these procedures is included in Chapter 12 of the FSP. Table 12-1 of the FSP provides typical requirements necessary for control of field instrumentation.

12.2 Laboratory Instruments

The EMSL QAP (Appendix C) presents the discussion of the laboratory's routine preventive maintenance program, which will be conducted to minimize the occurrence of instrument failure and other system malfunctions. All laboratory instruments will be maintained in accordance with manufacturer's specifications and the requirements of the specific method employed. This maintenance will be carried out on a regular, scheduled basis and will be documented in the laboratory instrument service logbook for each instrument. Emergency repair or scheduled manufacturer's maintenance will be provided under a repair and maintenance contract with factory representatives. Table 12-1 provides typical maintenance items necessary for the subcontract lab to perform.

13.0 Performance and System Audits

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the FSP and QAPP. Audits of laboratory activities will include both internal and external audits. EMSL's QAP (Appendix C) provides a discussion of performance and system audits.

13.1 External Laboratory Audits

The NJDEP/USEPA conducts on-site audits and validates laboratories on a regular basis. This independent on-site systems audit in conjunction with performance evaluation samples (performance audits) qualify laboratories to perform NJDEP/USEPA environmental analysis every 18 months.

These system audits include examining laboratory documentation of sample receiving, sample log-in, sample storage, COC procedures, sample preparation and analysis, instrument operating records, etc. Performance audits consist of sending performance evaluation samples to NJDEP/USEPA laboratories for on-going assessment of laboratory precision and accuracy. The analytical results of the analysis of performance evaluation samples are evaluated by NJDEP/USEPA to ensure that laboratories maintain an acceptable performance.

Additionally, external audits will be performed by the contractor's Program Chemist to ensure the analytical laboratory's capabilities initially demonstrated for the NJDEP/USEPA audit, are still properly implemented. Similar audit issues and the use of a NJDEP/USEPA approved checklist will provide documentation of the audit action.

13.2 Internal Laboratory Audits

The Laboratory QA Officer as directed in the laboratory QA plan will conduct internal performance and system audits of their analytical laboratory. These system audits will include examination of laboratory documentation of sample receiving, sample log-in, sample storage, COC procedures, sample preparation and analysis, instrument operating records, etc. Internal performance audits are also conducted on a regular basis. Single-blind performance samples will be prepared and submitted along with project samples to the laboratory for analysis. The Laboratory QA Officer will evaluate the analytical results of these single-blind performance samples to ensure that the laboratory maintains acceptable performance.

Additional audits of laboratories may be planned and budgeted within specific NJDEP/USEPA task scopes. These project-specific laboratory performance review audits would be conducted by the contractor at the direction of and in conjunction with the NJDEP/USEPA, when requested.

14.0 Quality Assurance Reports to Management

14.1 Daily Chemical Quality Control Reports

During the active field investigation activities performed for this project, the contractor will prepare Daily Chemical Quality Control Reports (DCQCRs), which will be signed and dated by the contractor Project Chemist or designee. An example of the DCQCR format to be used by the contractor is illustrated on Figure 14-1. These reports will be submitted to the NJDEP/USEPA Technical/Project Manager on an as needed basis. The contents of each DCQCR will include a summary of activities performed at the project site, weather information, results of Contractor Chemical Quality Control (CCQC) activities performed including field instrument calibrations, departures from the approved Work Plan problems encountered during field activities, and any instructions received from government personnel. Any deviations that may affect the project data quality objectives will be immediately conveyed to the appropriate NJDEP/USEPA Manager. EMSL's QAP provides a discussion of QA Reports to the Management.

14.2 Quality Assurance Reports

Each laboratory will provide LORs and analytical QC summary statements (case narratives) with each data package. All COC forms will be compared with samples received by the laboratory and a LOR will be prepared and sent to the contractor describing any differences in the COC forms and the sample labels or tags. All deviations will be identified on the receiving report such as broken or otherwise damaged containers. This report will be forwarded to the contractor within 24 hours of sample receipt and will include the following: a signed copy of the COC form; itemized sample numbers; laboratory sample numbers; cooler temperature upon receipt; and itemization of analyses to be performed.

Summary QC statements will accompany analytical results as they are reported by the laboratory in the form of case narratives for each sample delivery group.

Any departures from approved plans will receive prior approval from the NJDEP/USEPA District Project Manager and will be documented with field change orders. These field change orders will be incorporated into the project evidence file.

The contractor will maintain custody of the project evidence file and will maintain the contents of files for this project, including all relevant records, reports, logs, field logbooks, pictures, subcontractor reports, correspondence, and COC forms, until this information is transferred to

the NJDEP/USEPA Project Manager. These files will be stored under custody of the contractor Project Manager. Analytical laboratories will retain all original analytical raw data information (both hard copy and electronic) in a secure, limited access area and under custody of the laboratory Project Manager.

14.3 Quality Control Summary Reports

At the conclusion of field investigation activities and laboratory analysis, the contractor, in addition to any review conducted by the laboratory, will perform its own validation of the submitted data. This activity will include assignment of flags to data, documentation of the reason(s) for the assignments, and description of any other data discrepancies. The contractor will then prepare a QCSR, which will be included as an appendix to the final report. This report will be submitted to the NJDEP/USEPA District Project Manager as determined by the project schedule. The contents of the QCSR will include data validation documentation and discussion of all data that may have been compromised or influenced by aberrations in the sampling and analytical processes. Both field and laboratory QC activities will be summarized, and all DQCR information will be consolidated. Problems encountered, corrective actions taken, and their impact on project DQOs will be determined.

The following are examples of elements to be included in the QCSR, as appropriate:

- Laboratory QC evaluation and summary of the data quality for each analytical type and matrix. Part of the accuracy, precision, and sensitivity summarized in the data quality assessment.
- Field QC evaluation and summary of data quality relative to data usability. Part of the accuracy, precision, and sensitivity summarized in the data quality assessment.
- Overall data assessment and usability evaluation.
- DCQCR consolidation and summary.
- Summary of lessons learned during project implementation.

Specific elements to be evaluated within the QCSR include the following:

- Sample results
- Field and laboratory blank results
- Laboratory control sample percent recovery (method dependent)
- Sample matrix spike percent recovery (method dependent)

- Matrix spike/matrix spike duplicate or sample duplicate RPD (method dependent)
- Analytical holding times
- Surrogate recovery, when appropriate.

15.0 References

U. S. Environmental Protection Agency (USEPA) 1985. <u>NEIC Policies and Procedures</u>, EPA-300/9-78DDI-R, Revised June.

USEPA 1991. <u>Interim Guidelines and Specifications for Preparing Quality Assurance Project</u> Plans,

QA/R5, revised October 1999

USEPA 1993a. Data Quality Objectives Process, EPA-540-R-93-071, September.

USEPA 1993b. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition, Revision 1, Update 1.

USEPA 1994a. EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, EPA QA/R-5, January.

USEPA 1994b. <u>EPA Contract Laboratory Program National Functional Guidelines for Organic</u> Data Review, EPA-540/R-94/012, February.

USEPA 1994c. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94/013, February.

USACE (U. S. Army Corps of Engineers) 1994. Requirements for the Preparation of Sampling and Analysis Plans, EM 200-1-3, September.

USACE 1998. Chemical Data Quality Management for Hazardous, Toxic, Radioactive Waste Remedial Activities, ER 1110-1-263, April.

USACE 1998b. Technical Project Planning Process, EM-200, 1-2.

USEPA 1999. Interim Final QA/R-5, EPA Requirements For QAPPs For Environmental Data Operations.

USEPA 1998. QA/G-5, EPA Guidance For QAPPs.

USACE October 10, 1997. EM-200-1-6, Chemical Quality Assurance for HTRW Projects.

Tables

TABLE 3-1 Sediment DQI Summary for OU1

Data Use	Sample Type	Analytical Method	Precision; Field Duplicates RPD ¹	Precision; Laboratory Duplicates RPD ¹	Accuracy ² ; LCS, %R ³	Completeness ⁴
Confirmation that remedial action objectives have met the minimum requirements	Discrete or Composite	Bulk Asbestos (PLM)	TBD	NA	NA	90%

¹ Relative Percent Difference (RPD); assess values greater than 5 times the reporting limit; otherwise use ±3 times RL as control ² Accuracy Range provided; actual parameter recoveries provided in method SOP ³ Percent Recovery (%R)

⁴Completeness based upon the results of data validation

TABLE 3-2 Ground Water/Surface Water DQI Summary for OU1

Data Use	Sample Type	Analytical Method	Precision; Field Duplicates RPD	Precision; Laboratory Duplicates RPD	Accuracy ² ; LCS, %R ³	Completeness ⁴
Confirmation that remedial action objectives have met the minimum requirements	Discrete or Composite	Bulk Asbestos (TEM)	TBD	NA	NA	90%

¹ Relative Percent Difference (RPD); assess values greater than 5 times the reporting limit; otherwise use ±3 times RL as control ² Accuracy Range provided; actual parameter recoveries provided in method SOP

³ Percent Recovery (%R)

⁴Completeness based upon the results of data validation

TABLE 4-1 Container Requirements for Sediment Samples for OU1

Analytical Group	Container	Minimum Sample Size	Preservative	Holding Time
Bulk Asbestos				
(PLM)	250 ml glass	100 g	None	14 days from collection

TABLE 4-2 Container Requirements for Water Samples for OU1

Analytical Group	Container	Minimum Sample Size	Preservative	Holding Time
Bulk Asbestos (TEM)	1L Plastic	Full	None	2 weeks

Table 9-1
Statistical Calculations OU-1

Statistic	Symbol	Formula	Definition	Use
Mean	$\frac{1}{x}$	$\left(\begin{array}{c} \sum_{i=1}^{n} x_{i} \\ n \end{array}\right)$	Measure of central tendency	Used to determine the average value of multiple measurements
Standard Deviation	S	$\sqrt{\left(\frac{\sum_{i=1}^{n} (x_i - \overline{x})^2}{n-1}\right)}$	Measure of the relative scatter of the data	Used in calculating variation of measurements
Relative Standard Deviation	RSD	$\left(S/\overline{x}\right) \times 100$	Relative standard deviation adjusts for the magnitude of observations	Used to assess the precision parameter for replicate results
Percent Difference	%D	$\left(\frac{x_1-x_2}{x_1}\right)\times 100$	Measure of the difference between two observations	Used to assess the accuracy parameter
Relative Percent Difference	RPD	$\left(\frac{x_1 - x_2}{\left(x_1 + x_2\right) \div 2}\right) \times 100$	Measure of variability that adjusts for the magnitude of observations	Used to assess the analytical precision of duplicate measurements
Percent Recovery	% R	$\left(\frac{x_{measured}}{x_{true}}\right) \times 100$	Recovery of spiked compounds in control sample (LCS)	Used to assess the accuracy parameter
Percent Recovery	% R	$\frac{\left(x_{s}-x_{u}\right)}{x_{a}} \times 100$ where: x _s is the value of the spiked sample, x _u is the value of the unspiked sample, x _u is amount spiked into the sample	Recovery of spiked compounds in the sample matrix	Used to assess matrix effects and precision between the MS and MSD

Table 11-1 Summary of Analytical Hardcopy Data Deliverables

Method requirements	Deliverables
Requirements for all methods:	
Holding time information and methods requested	Signed chain-of-custody forms
Discussion of laboratory analysis, including any	
laboratory problems	Case narratives
Organics: HPLC analysis	
Sample results	CLP Form 1 or equivalent
Surrogate recoveries	CLP Form 2 or equivalent
Matrix spike/spike duplicate data	CLP Form 3 or equivalent
Method blank data	CLP Form 4 or equivalent
Initial calibration data	CLP Form 6 or equivalent
If calibration factors are used	A form listing each analyte, the concentration of each
	standard, the relative calibration factor, the mean
	calibration factor, and %RSD
Calibration curve if used	Calibration curve and correlation coefficient
Continuing calibration data	CLP Form 9 or equivalent
Positive identification (second column confirmation)	CLP Form 10 or equivalent
Metals Sample results	CI P Form 1 or equivalent
- Sample results	CLP Form 1 or equivalent CLP Form 2 or equivalent dates of analyses and
	CLP Form 2 or equivalent, dates of analyses and
Sample results Initial and continuing calibration	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor
Sample results Initial and continuing calibration Method blank	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor CLP Form 3 or equivalent and dates of analyses
Sample results Initial and continuing calibration Method blank ICP interference check sample	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor CLP Form 3 or equivalent and dates of analyses CLP Form 4 or equivalent and dates of analyses
Sample results Initial and continuing calibration Method blank ICP interference check sample Spike sample recovery	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor CLP Form 3 or equivalent and dates of analyses CLP Form 4 or equivalent and dates of analyses CLP Form 5A or equivalent
 Sample results Initial and continuing calibration Method blank ICP interference check sample Spike sample recovery Post-digestion spike sample recovery for ICP metals 	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor CLP Form 3 or equivalent and dates of analyses CLP Form 4 or equivalent and dates of analyses CLP Form 5A or equivalent CLP Form 5B or equivalent
Sample results Initial and continuing calibration Method blank ICP interference check sample Spike sample recovery Post-digestion spike sample recovery for ICP metals Post-digestion spike for GFAA	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor CLP Form 3 or equivalent and dates of analyses CLP Form 4 or equivalent and dates of analyses CLP Form 5A or equivalent CLP Form 5B or equivalent CLP Form 5B or equivalent
 Sample results Initial and continuing calibration Method blank ICP interference check sample Spike sample recovery Post-digestion spike sample recovery for ICP metals 	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor CLP Form 3 or equivalent and dates of analyses CLP Form 4 or equivalent and dates of analyses CLP Form 5A or equivalent CLP Form 5B or equivalent CLP Form 5B or equivalent CLP Form 6 or equivalent
Sample results Initial and continuing calibration Method blank ICP interference check sample Spike sample recovery Post-digestion spike sample recovery for ICP metals Post-digestion spike for GFAA Duplicates	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor CLP Form 3 or equivalent and dates of analyses CLP Form 4 or equivalent and dates of analyses CLP Form 5A or equivalent CLP Form 5B or equivalent CLP Form 5B or equivalent CLP Form 6 or equivalent
Sample results Initial and continuing calibration Method blank ICP interference check sample Spike sample recovery Post-digestion spike sample recovery for ICP metals Post-digestion spike for GFAA Duplicates LCS	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor CLP Form 3 or equivalent and dates of analyses CLP Form 4 or equivalent and dates of analyses CLP Form 5A or equivalent CLP Form 5B or equivalent CLP Form 5B or equivalent CLP Form 6 or equivalent CLP Form 7 or equivalent CLP Form 7 or equivalent that includes acceptable rangor window
Sample results Initial and continuing calibration Method blank ICP interference check sample Spike sample recovery Post-digestion spike sample recovery for ICP metals Post-digestion spike for GFAA Duplicates	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor CLP Form 3 or equivalent and dates of analyses CLP Form 4 or equivalent and dates of analyses CLP Form 5A or equivalent CLP Form 5B or equivalent CLP Form 5B or equivalent CLP Form 6 or equivalent CLP Form 7 or equivalent CLP Form 7 or equivalent that includes acceptable rang or window CLP Form 8 or equivalent
Sample results Initial and continuing calibration Method blank ICP interference check sample Spike sample recovery Post-digestion spike sample recovery for ICP metals Post-digestion spike for GFAA Duplicates LCS Standard additions (when implemented)	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor CLP Form 3 or equivalent and dates of analyses CLP Form 4 or equivalent and dates of analyses CLP Form 5A or equivalent CLP Form 5B or equivalent CLP Form 5B or equivalent CLP Form 6 or equivalent CLP Form 7 or equivalent CLP Form 7 or equivalent that includes acceptable rangor window
Sample results Initial and continuing calibration Method blank ICP interference check sample Spike sample recovery Post-digestion spike sample recovery for ICP metals Post-digestion spike for GFAA Duplicates LCS Standard additions (when implemented) Holding times Run log	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor CLP Form 3 or equivalent and dates of analyses CLP Form 4 or equivalent and dates of analyses CLP Form 5A or equivalent CLP Form 5B or equivalent CLP Form 5B or equivalent CLP Form 6 or equivalent CLP Form 7 or equivalent CLP Form 8 or equivalent cLP Form 8 or equivalent CLP Form 8 or equivalent CLP Form 13 or equivalent
Sample results Initial and continuing calibration Method blank ICP interference check sample Spike sample recovery Post-digestion spike sample recovery for ICP metals Post-digestion spike for GFAA Duplicates LCS Standard additions (when implemented) Holding times Run log CLP Contract laboratory program	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor CLP Form 3 or equivalent and dates of analyses CLP Form 4 or equivalent and dates of analyses CLP Form 5A or equivalent CLP Form 5B or equivalent CLP Form 5B or equivalent CLP Form 6 or equivalent CLP Form 7 or equivalent CLP Form 8 or equivalent cLP Form 8 or equivalent CLP Form 8 or equivalent CLP Form 13 or equivalent
Sample results Initial and continuing calibration Method blank ICP interference check sample Spike sample recovery Post-digestion spike sample recovery for ICP metals Post-digestion spike for GFAA Duplicates LCS Standard additions (when implemented) Holding times Run log CLP [] contract laboratory program HPLC - high performance liquid chromatography	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor CLP Form 3 or equivalent and dates of analyses CLP Form 4 or equivalent and dates of analyses CLP Form 5A or equivalent CLP Form 5B or equivalent CLP Form 5B or equivalent CLP Form 6 or equivalent CLP Form 7 or equivalent CLP Form 8 or equivalent that includes acceptable rang or window CLP Form 8 or equivalent CLP Form 13 or equivalent
Sample results Initial and continuing calibration Method blank ICP interference check sample Spike sample recovery Post-digestion spike sample recovery for ICP metals Post-digestion spike for GFAA Duplicates LCS Standard additions (when implemented) Holding times Run log CLP	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor CLP Form 3 or equivalent and dates of analyses CLP Form 4 or equivalent and dates of analyses CLP Form 5A or equivalent CLP Form 5B or equivalent CLP Form 5B or equivalent CLP Form 6 or equivalent CLP Form 7 or equivalent CLP Form 7 or equivalent that includes acceptable rang or window CLP Form 8 or equivalent CLP Form 13 or equivalent
Sample results Initial and continuing calibration Method blank ICP interference check sample Spike sample recovery Post-digestion spike sample recovery for ICP metals Post-digestion spike for GFAA Duplicates LCS Standard additions (when implemented) Holding times Run log CLP	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor CLP Form 3 or equivalent and dates of analyses CLP Form 4 or equivalent and dates of analyses CLP Form 5A or equivalent CLP Form 5B or equivalent CLP Form 5B or equivalent CLP Form 6 or equivalent CLP Form 7 or equivalent CLP Form 7 or equivalent that includes acceptable rang or window CLP Form 8 or equivalent CLP Form 13 or equivalent
Sample results Initial and continuing calibration Method blank ICP interference check sample Spike sample recovery Post-digestion spike sample recovery for ICP metals Post-digestion spike for GFAA Duplicates LCS Standard additions (when implemented) Holding times Run log CLP	CLP Form 2 or equivalent, dates of analyses and calibration curve, and the correlation coefficient factor CLP Form 3 or equivalent and dates of analyses CLP Form 4 or equivalent and dates of analyses CLP Form 5A or equivalent CLP Form 5B or equivalent CLP Form 5B or equivalent CLP Form 6 or equivalent CLP Form 7 or equivalent CLP Form 8 or equivalent that includes acceptable rang or window CLP Form 8 or equivalent CLP Form 13 or equivalent

Table 11-2 Standard Electronic Data Deliverables (ITEMS)

Comments:

- a. The lab should enter the lab sample number in both the project and lab sample number fields when the sample is a laboratory QC sample.
- b. The sample date and time are required for all samples. Additionally, sample purpose is required for laboratory QC samples.
- c. The code reported by the lab is assigned by IT Corp.
- d. A blank qualifier is assumed to be a positive detect.
- e. Retention time is required for Tentatively Identified Compounds (TICs) only. For Target compounds and surrogates, leave this field empty.
- f. The detection limit reported in this field is the actual detection limit that the lab experienced for the particular sample and analysis.
- g. The detection limit reported in this field is the limit for the method as reported in the literature.
- h. If the sample is not diluted, report a value of 1.
- i. These fields are intended to be used only by projects that eventually upload their data from ITEMS into IRPIMS.
- j. Sample Prep. Code and Extraction Date are required fields.
- k. Valid entries are Y/N.
- 1. Valid entries are 0-9. Zero (0) should be reported for normal sample results.

Table A: Results Types

Result Type	Category	Description
TRG	NF	Target parameter for analysis
TIC	NF	Tentatively Identified Compound
IS	LQ	Internal Standard added to the sample solution by the laboratory
SUR	LQ	Surrogate compound added to the sample solution by the laboratory

Table 11-2 Standard Electronic Data Deliverables (ITEMS)

Table B: Result Qualifiers

Qualifier	Qualifier Category	Non-detect Qualifier	Description
U	0	Y	Compound was analyzed for, but was not detected ([]Non-detect)
J	0	N	Estimated value, less than the CRQL
С	0	N	Pesticides only. Presence confirmed by GC/MS
В	0	N	Analyte found in both sample and blank
Е	0	N	Estimated; result greater than upper end of linear range
D	0	N	Dilution run. Initial run greater than upper end of calibrated range
A	0	N	Indicates the TIC is a suspected Aldol condensation product
х	0	N	Indicates manual modification of result or EPA qualifier.
JX	0	N	Result is less than SQL that would have been displayed for []U[].
В	I	N	Value less than CRDL, but greater than or equal to IDL
E	I	N	Value estimated due to interference
М	I	N	Duplicate injection precision greater than 20% (for GFAAS)
N	I	N	Sample spike QC not recovered within control limits.
S	1	N	Method of Standard Additions (MSA) used to quantitate result
w	I	Ņ	Post-digestion spike out of control (GFAAS)
*	I	N	Laboratory duplicate analysis not within control.
+	I	N	Correlation coefficient for MSA is less than 0.995
P	I	N	Method qualifier - ICP
Α	I	N	Method qualifier - Flame AA
F	I	N	Method qualifier - Furnace AA
CV	1	N	Method qualifier - Manual Cold Vapor
AV	I	N	Method qualifier - Automated Cold Vapor
NR	1	N	Method qualifier - Analyte was not required
С	I	N	Method qualifier - Manual Spectrophotometric

Table 11-2 Standard Electronic Data Deliverables (ITEMS)

Table C: Sample Purpose

Sample Purpose	Category	Description
BKS	LQ	Blank Spike
BLK	LQ	Blank
BSD	LQ	Blank Spike Duplicate
СВ	LQ	Calibration Blank
LCS	LQ	Laboratory Control Sample
LR	LQ	Laboratory Replicate
МВ	LQ	Method Blank
MS	LQ	Matrix Spike
MSD	LQ	Matrix Spike Duplicate
REG	NF	Regular Environmental Sample
AB	FQ	Ambient Blank (per HAZWRAP definition)
ER	FQ	Equipment Rinsate
FB	FQ	Field Blank (EPA definition)
FD	FQ	Field Duplicate
RD	FQ	Regulatory Duplicate (collected in the field by regulator)
SMQC	FQ	Source Material Quality Control
SPLT	FQ	Split Regular Sample (each half is sent to a different lab)
ТВ	FQ	Trip Blank

Table D: Sample Preparation Codes

Préparation Code	Description
CIT	Waste Extraction Test using sodium citrate
CONI	Confirmational Analysis - first run
CON2	Confirmational Analysis - second run
DION	Waste Extraction Test using deionized water
NORM	Normal Preparation consistent with analytical method

Table 12-1
Preventive Maintenance Requirements for Laboratory Instruments
OU-1

Instrument	Activity	Frequency
High Performance Liquid Chromatograph (HPLC)	Check / change degas gases Check / change guard column Check / replace pre-column frits Monitor UV lamp intensity Replace Column Check flows	Daily Weekly Weekly As needed As needed Weekly
Inductively Coupled Plasma Optical Emission Spectrometer (ICP or ICPOES)	Check gas flow Clean nebulizer Check torch Change tubing Check optics	Daily Weekly Weekly, or as needed Weekly, or as needed Annual service contract
Ovens	Temperature monitoring	Once daily
Refrigerators	Temperature monitoring	Once daily
Analytical Balances	Check pans and compartment Check alignment and calibration Cleaning/ Service	Prior to use Before every use Semi-anually

Figures



COOLER RECEIPT / CONDITION UPON RECEIPT CHECKLIST

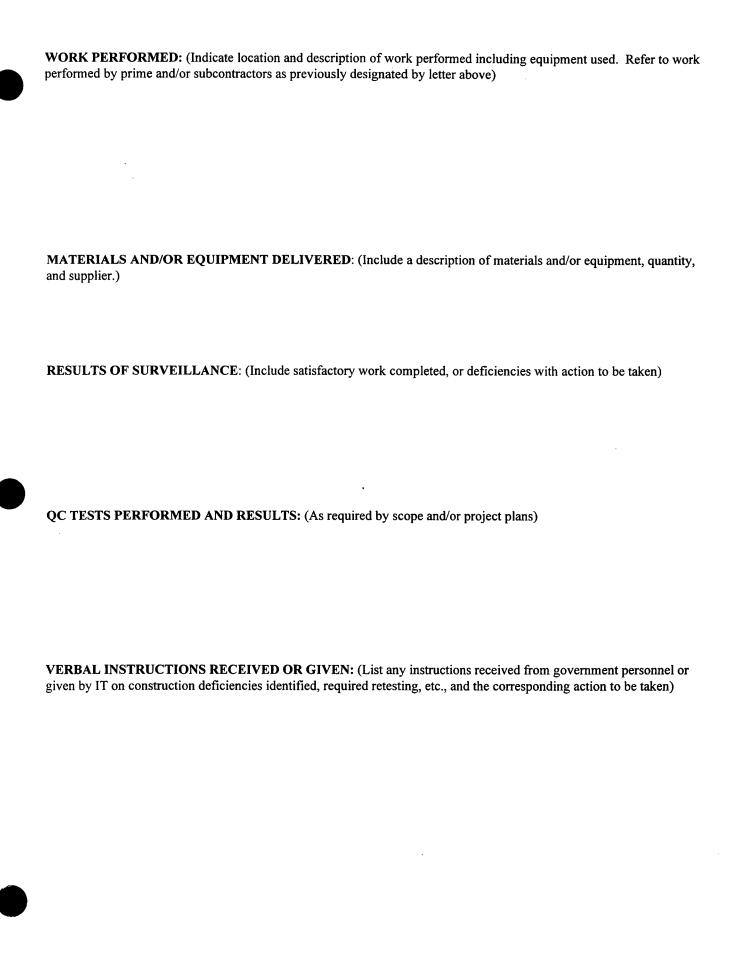
Client:_	Project:	SDG:		
To Be C	ompleted by Sample Custody:			
Sample Red	ceipt	Yes	No	NT A
Do	sample container labels match COC? (i.e. IDs, dates, ti	mes)	110	NA
Is th	the cooler temperature with $4\pm 2^{\circ}$ C?			
Wer	e the samples received with the prescribed preservative			
Wer	e custody seals intact/present on cooler/containers?			
Wer	e all samples listed on COC received?			
Wer	e all sample containers received intact?	-		
Wer	e water VOA vials received without headspace?			
Wer	e samples received in the appropriate containers?	-		-
Wer	e samples received with [] the QAPP holding time?			-
Were	e samples screened for radioactivity?			
Were	e client sample documents (COC, RFA) received?			
Was	the RFA/COC relinquished properly (signatures/dates)			
Are t	test parameters listed for all samples received?	·· —		
Are o	client specific QC requested/supplied?			
Is the	e date and time of sample collection noted on COC?			
Is the	e client/project name clearly identifiable?			
Sample Re	ceiving Representative:	Date	:	
To Be Co Sample Chec	mpleted by Project Management:			
_		Yes	No	NA
Was t	ient IDs on Log-in report match with RFA/COC IDs?			
vv as i	the COC/RFA signed and dated upon receipt			
Ara a	servative check noted on the COC?			
Ale C	ooler temperature & custody seals condition noted?			
Were	test parameters assigned correctly?			
were	correct analytical and report due dates assigned?			
was t	he proper report format indicated?			
Are cl	ient assigned QC samples correctly defined?			
is ther	e a contract number or PO for the work?			
Project Man	nagement Representative:	Date:		

IZCDOLL 140.	Re	port	No.
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Date:

Quality Control Summary Report

Project Name: Mil	lington Dump OU1 S	Site			
Contract No. DACW41-94-D9013			IT Project No	o.:	780601
Delivery Order No. 009			Project Location:		Millington, NJ
WEATHER:	() Clear	() Par	tly Cloudy	(X) Cloudy
Temperature:	High ^O F	Low	°F	Wind:	light
Site Conditions:					
PRIME CONTRA	CTOR/SUBCONTE	RACTORS A	AND AREAS C	OF RESPO	ONSIBILITY/LABOR COUNT:
a.					
b.					
c.					
d.					
e.					
f.					
g.					
h.					
Operating plant eq	quipment with hours	s worked, id	le or down tim	e for repa	ir:
a.					
b.					
c.					
d.					
e.					



CHANGED CONDITIONS/DELAYS/CONFLICTS ENCOUN [i.e.scope and/or project plans], any delays to the project attributal	NTERED: (List any conflicts with the delivery order ble to site and weather conditions, etc.)
	. ,
MEETINGS: (List the meetings, i.e., Health and Safety, Site Open	rations, Cost/Schedule, etc.)
VISITORS: (List name and affiliation)	
REMARKS: Any additional information pertinent to the project (ie Submittals reviewed)
CONTRACTOR'S VERIFICATION: I certify that the above reporteresentative, have inspected all work performed this day by the Predetermined that all materials, equipment and workmanship are in strong be noted above.	ime Contractor and each subcontractor and have
IT Quality Control System Manager	Date

Appendix A

CENWK-EC-EF Data Quality Evaluation Guidance July 26, 1999

This guidance is to be used for all chemical data quality evaluations performed by this district. It is based on criteria presented in SW-846 and provides guidance concerning data quality in areas not specifically covered by that publication. Finally, this guidance provides stricter adherence to criteria than does SW-846.

The specific order of review is not absolute. The reviewer may choose the order which is most useful. Although some review items may require that a sample result be not usable, the reviewer must complete all items in their analysis of a sample result.

The review checklists may be used for sample delivery groups, individual samples, or some other grouping.

Separate sections are presented for inorganic and organic analysis.

Applicability. This Guidance is to be used for all Kansas City District evaluation of chemical data quality. The criteria for acceptable data and usable data will be defined in both the Scope of Work (SOW) and the Quality Assurance Project Plan (QAPP). This Guidance is designed for determination of acceptability of the data, not for usability. Usability will be a separate determination. These terms are defined in the next section and in the SOW.

Purpose. The number and diverse backgrounds of all chemists involved in data evaluation tends to result in conflicting conclusions about the acceptability of the data. This standard was therefore necessary and was developed through agreement of all District chemists. It is the purpose of this standard to reduce variability in data quality evaluation and allow all organizations associated with this District to understand the basis this District uses in evaluating data quality.

Data shall be evaluated based on criteria listed in the SW-846 methods. For non-SW-846 methods, data shall be evaluated according to the criteria specified in the QAPP. Data shall be rejected if any part of these criteria have not been met, and no documented corrective actions as described in the QAPP have been taken. If the corrective action(s) corrected the problem, all

data generated under the corrected conditions may be reported without qualification. If appropriate corrective actions have been taken, but QC criteria still could not be met, data shall be flagged (provided that the corrective action taken was that described in the SW-846 methodology or the QAPP). Such data shall be considered acceptable data. Quality data is data obtained in a sample batch for which all quality control criteria were met. Data will be further evaluated by comparing the A-E's data to duplicate data which will be available from the USACE QA laboratory.

It should be noted that corrective actions are required for each failure to meet established QC criteria. When such corrective action is not described in the data package, the following alternatives should be selected (listed in order of preference):

- a. Contact the lab to determine why no corrective action was taken.
- b. Reject the batch if the QC exceedence is batch QC or the particular sample if the exceedence is sample-specific (i.e., surrogates, detection limit, etc.). USACE may accept batch if, in the reviewer's judgement, the exceedence is minimal.
- Make a comment in the data review that the data package is missing corrective actions and the necessary review cannot be completed without it.

It should also be noted that this Guidance does not evaluate all the quality control checks listed in the attached tables. Additional QC data will be evaluated when a decision on the data quality is in doubt.

NOTE: The criteria for determining the acceptability of the data as described in this Guidance are subject to exceptions. Specific circumstances may permit a detemination that some data are acceptable or not acceptable even though the literal interpretation of the Guidance would indicate otherwise. Such circumstances must be clearly and completely documented.

Explanation of Terms

Acceptable Data. Acceptable data are the best data the laboratory is able to produce within the confines of the Scope of Work and the approved QAPP. Acceptable data will be contract-compliant and not subject to further laboratory work. Under this definition, acceptable data may still be subject to rejection.

Estimated Data. Data which failed to meet quality control criteria even after corrective actions have been taken. If, in the chemist's judgement this failure is sufficiently severe, this data may actually be rejected.

Quality Data. Data which has met all of the quality control criteria.

Rejected Data. Reported analytical data which the chemist believes does not reliably reflect the actual amount of analyte in the sample and/or it does not fit the definition of acceptable data.

Usable Data. Data which are considered usable for the project. Only under rare circumstances would rejected data be considered usable. The determination of the usability of the data is not considered in the implementation of this Guidance.

Organic Data Evaluation

Refer to Organic Data Evaluation Checklist and Attached QC Criteria Tables

- 1. Chain-of-Custody. Determine if chain-of-custody (CoC) form is present and properly signed. Inspect to determine if any problems were noted with the form by the laboratory upon receipt of the samples. Problems which cast doubt on the identity of certain sample will result in an automatic rejection of those samples from the same cooler unless all sample results appear below detection limits.
- 2. Preservation. Determine if cooler receipt form is present and signed by the laboratory. Determine if sample is preserved and sample integrity has been maintained according to the QAPP. Samples which were improperly preserved may be rejected. Qualify as estimated (J code) any samples requiring cooling as part of their preservation whose temperature was in the range of 6-9 °C.
- 3. Requested Analysis. Determine if the CoC-requested analyses were performed.
- 4. Holding Times. Determine if extraction and analysis holding times for samples were met. If a holding time is missed, then samples will be rejected.
- 5. For aqueous volatile organic samples, make sure Blanks. that an associated trip blank was present and analyzed in the same manner as the samples. Field sample results will be qualified as undetected (U) if the concentration in the sample is less than five times the concentration in the associated laboratory method blank, instrument blank, rinsate blank, or trip blank, with the detection limit reported the same as the analytical result. For common laboratory compounds such as methylene chloride, acetone, 2butanone, and common phthalate esters (or additional/different contaminants as indicated from laboratory's data), a result will be qualified as described above if the sample concentration is less than ten times the concentration in the laboratory blank, with the detection limit reported as the analytical result for that sample. Analytical results above these limits will be reported as Alteration of sample analytical results due to blank concentrations is not permissable.

- 6. Review LCS/LCSD percent recoveries and relative percent differences (RPD). The LCS and LCSD should contain only the analytes used for the matrix spikes or the analytes of interest for the site. If outside of accuracy and precision limits set by the laboratory (below LCL (lower control limit) or above UCL (upper control limit)), reject data for that batch. If MS/MSD criteria are met and the LCS/LCSD are not, the analytical data for that sample (with the MS/MSD results) only is acceptable.
- 7. Review blind field quality control (QC) duplicates. The degree of agreement between these duplicates is to be used in conjunction with all of the remaining quality control results as an aid in the decision as to the overall quality of the data. Data are not to be qualified due to QC duplicates alone. To determine the level of agreement between the duplicates, the following guidelines have been established:
 - a. For all analyses in water matrices, data should be considered in agreement if results are within a factor of two of each other. Data between a factor of two and three of each other should be considered as a minor discrepancy and data greater than a factor of three should be considered a major discrepancy.
 - b. For soil analyses, data should be considered in agreement if results are within a factor of four of each other. Data between a factor of four and five of each other should be considered as a minor discrepancy, and data greater than a factor of five should be considered a major discrepancy. Note that the comparison criterion for soil samples is based on samples homogenized in the field.
- 8. Review surrogate results. Recoveries in each field sample and laboratory QC sample will be examined to see if they are within acceptable range. For surrogates <LCL or >UCL for which corrective actions have been performed, the associated analytes in that sample will be "J" qualified if:
 - a. Two base/neutral, two acid-extractable or two
 pesticide/PCB surrogates are outside above criteria in a
 SVOC sample;
 - b. One surrogate from any other organic sample is outside above criteria.
 - If no required corrective actions have been performed, the

corresponding sample(s) will be rejected. If surrogate recoveries as described above are equal to or less than 10%, sample results will be rejected (R).

- 9. Review the matrix spike/matrix spike duplicates (MS/MSD). If the recovery and/or RPD values are out of range and corrective actions solved the problem, all data generated under the corrected conditions can be accepted without qualification. If the recovery and/or RPD values are out of range and corrective actions did not solve the problem, then sample results should at least be flagged as estimated (J). If corrective actions were not taken, sample results should be rejected for associated analytes in the batch.
- 10. Examine sample quantitation limits to determine if project-required quantitation limits are met. Make note if out of range. Laboratory must supply cause(s) as to why QLs are above criteria. If causes are uncorrectable, no further action is necessary. If correctable (such as sample cleanup as recommended in the method, or running undiluted samples), resampling and reanalysis may be required.
- 11. When available, the data comparison tables in the QAR (Quality Assurance Report from MR lab) are checked for comparability of the QA splits with the contractor's samples. Discrepancies are judged in the same manner as the blind duplicates (see par. 7 in this section).

If the QAR is not available and a data judgement must be made, the QA analytical results (this is not the QAR) should be matched with the contractor's corresponding sample. If sample numbers don't match, the contractor should be contacted for the matching sample numbers.

- 12. For situations where an obvious judgement on data quality cannot be made, the following minimum alternatives could be used:
 - a. Ask the lab for additional data, such as chromatograms, calibration curves, etc. needed for the decision. The SOW normally states that such a request can be made. If the SOW has no such statement, rely on the lab's spirit of cooperation to get the needed information.
 - b. For questions where one of the questionable data points includes a sample which was split to the QA lab, the QA lab should be requested to contact the primary (QC) lab. The project chemist should be part of the

discussion between the contractor lab and the QA lab.

Inorganic Data Evaluation

Refer to Inorganic Data Evaluation Checklist and Attached QC Tables.

- A. Graphite Furnace, and Cold Vapor 7000 Series Methods Follow steps 1 through 4 in the Organic Data Review section.
- 5. Review LCS (and LCSD percent recovery if available). If outside of accuracy limits, reject data for the corresponding analytes in that batch. If MS is within criteria, accept that analyte for only that sample in the batch.
- 6. Blanks. Field sample results will be qualified as undetected (U) if the concentration in the sample is less than five times the concentration in the associated laboratory method blank, instrument blank, reagent, calibration, or rinsate blank with the detection limit reported numerically equal to the analytical result. Analytical results above these limits will be reported as is. Alteration of sample analytical results due to blank concentrations is not permissable.
- 7. Review blind field quality control (QC) duplicates. The degree of agreement between these duplicates is to be used in conjunction with all of the remaining quality control results as an aid in the decision as to the overall quality of the data. Data are not to be qualified due to QC duplicates alone. To determine the level of agreement between the duplicates, the following guidelines have been established:

For all analyses in a water matrix and analysis in soil, data should be considered in agreement if results are within a factor to two of each other. Data between a factor of two and three of each other should be considered as a minor discrepancy and data greater than a factor of three should be considered a major discrepancy. Note that the comparison criteria for soil samples are based on samples homogenized in the field.

8. Review matrix spike/matrix duplicate (MS/MD) data. If the recovery is outside criteria all corresponding analytes are qualified as at least "J" if corrective actions have been taken but recovery is still outside criteria; otherwise data

are rejected (see attached tables).

- 9. Examine sample method detection limits to determine if project required method detection limits are met. Make note if out of range. Laboratory must supply cause(s) as to why QLs are above criteria. If causes are uncorrectable, no further action necessary. If correctable (such as sample cleanup as recommended in method), reanalysis and possibly resampling and reanalysis are required.
- 10. Interference Tests. Serial dilution (SD), recovery test, and method of standard additions (MSA) are all interrelated. The flow chart in Figure 1 indicates under what circumstances the data should be rejected, qualified as estimated, or accepted without qualification. If the QAPP provides an acceptable equivalent to the interference tests described below, the methods of evaluating the substituted test(s) will be determined at that time.

If %D between diluted value and the original value is >±10%, recovery test and/or MSA (Method of Standard Additions) must meet criteria. Accept data with qualification if recovery test or MSA meets criteria. If one or both of these procedures fail criteria, professional judgement will be used based on the extent of the failure(s). Generally, if MS (matrix spike) is acceptable, the analyte(s) for all other samples in the batch are acceptable and professional judgement is required only on the sample that failed the serial dilution. If MS also failed for the analyte(s), said analyte(s) will be rejected for all samples in that batch. If procedures were not run results for that analyte are rejected for that batch. This, and the following two steps narratively describe what the chart is stating.

- a. Serial Dilution. Should be run on sample which will theoretically be 5 times above method detection limit after a 5-fold dilution.
- b. Recovery Test. Should be run only on sample that either failed dilution test or on samples where the dilution test is not applicable¹, after sample is spiked at approximately 5 times the original concentration or 20 times the MDL. If not run and MSA not run, analyte(s) will be rejected for that batch. Percent recovery should be >85% AND <115% for results to be acceptable.

 $^{\rm 1}$ Seldom will a single sample contain all the metals of concern at 5 X detection level or above. Therefore, although the dilution test was conducted, it was not applicable for those metals. Regardless, recovery test should be run on that sample for those metals.

- c. Method of Standard Additions. If MSA run and met criteria, results of the MSA will be given as the analytical result with no qualification. If recovery test passes, result for that analyte will be "J" qualified for all samples in that batch. If this test is run because of failure of serial dilution and/or recovery test, MSA failure will call for professional judgement based in the magnitude of the failures. No further corrective action necessary.
- 11. If the reviewer suspects instrument calibration errors then calibration information should be reviewed in-depth. Consult QAPP for details of review. Acceptability of data should be reconsidered if uncorrected calibration errors occurred.
- B. Inductively Coupled Argon Plasma Method 6010Follow steps 1 to 4 in sec. A of the Inorganic Data Review.
- 5. Blanks. See Inorganics Data Review, sec. A.6.
- 6. Review blind field quality control and lab duplicates. See Inorganics Data Review, sec. A.7.
- 7. Review matrix spike/matrix duplicate (MS/MD) data. If the recovery is outside criteria, all corresponding analytes are qualified as "J" if corrective actions have been taken; otherwise data are rejected (see attached tables).
- 8. Examine sample detection limits to determine if projectrequired quantitation limits are met. Make note if out of
 range. Laboratory must supply cause(s) as to why QLs are
 above criteria. If causes are uncorrectable, no further
 action necessary. If correctable (such as sample cleanup as
 recommended in method), reanalysis and possibly resampling
 and reanalysis are required.
- 9. Serial Dilution. If interference is suspeced and the five-fold dilution analysis result is $> \pm 10\%$ of the original determination, data for that analyte in that sample only is qualified as estimated or rejected, depending on the extent of the deviation, and the acceptability of the matrix spike for the same analyte(s). If matrix spike was acceptable (recoveries in the range 75% to 125%) and run on the same sample as the serial dilution, the sample result will be qualified as estimated (J). If matrix spike was outside as

- well (whether or not MS run on the same sample), the affected analyte(s) will be rejected for all samples in that batch or qualified as estimated (J) if corrective action is taken but is still outside criteria. Although not required by the method, an MSA is highly recommended. Should an MSA be present, the flow chart on the previous page will be followed.
- 10. Intra-element Correction Factors and Interference Check Sample Requirements. If serious interference is suspected, calibration information (ICV, CCV, ICSA, ICSB, etc.) should be reviewed in depth. Contact the laboratory for additional information.

Miscellaneous Methods

As seen from the attached table, there is a large selection of methods which could possibly be used on any site. Though the selection is large, it will still not cover all methods which may be used by USACE. The data evaluation will follow the preceeding guidances when similar quality controls are required from the method.

Organic Data Evaluation Checklist

Project Name	
Project Cost Key	
Site	
Laboratory	
Laboratory Report Number	·
Analysis Type	
Sample Matrix	
Date Review Initiated/USACE Chemist's Initials	
Date Review Completed/USACE Chemist's Initials	

		T	T		
	Review Item	NA	Reviewed	Qualified	Comments
Chain-	of-Custody				
Cooler Tempe	Receipt Form—Sample erature/Preservation				
Reque	sted Analyses Completed				
Holdin	g Times				
Trip Bl	anks				
LCS Re	esults				
Method	Blanks				
Equipm	ent Rinsate Blanks				
Field Du	plicates				
Surroga	tes				
MS/MSI	D REC Results			-	
MS/MS[O RPD Results				
Project (Quantitation Limits				
					·
I:\MASTERS\	VALID1.TAB				

Inorganic Data Evaluation Checklist

Project Name	
Project Cost Key	
Site	
Laboratory	
Laboratory Report Number	
Analysis Type	
Sample Matrix	
Date Review Initiated/USACE Chemist's Initials	
Date Review Completed/USACE Chemist's Initials	

	Table 1		T	
Review Item	NA	Reviewed	Qualified	Comments
Chain-of-Custody				
Cooler Receipt Form-Sample Temperature/Preservation				
Requested Analyses Completed				
Holding Times				
LCS Results				·
Method Blanks				
Equipment Rinsate Blanks				
Field Duplicates				
MS/MD REC Results				
MS/MD RPD Results				
Project Quantitation Limits				
:\MASTERS\VALID2.TAB				

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA 160.1 160.2	Total Suspended Solids Total Dissolved Solids	Dissolved Duplicate 1 for every 10 field can		% RPD ≤ 35%	Review lab QC data to determine if they are in control. If not, qualify data. Use data to evaluate proper collection procedures were followed. If not, determine further
	Rinsate (c) 1 for ever collected LABORATORY OC:		1 for every 10 field samples collected	Less than reported detection limits	corrective action. Qualify data.
		Calibration of the instrument	According to the instrument service manual	All measures must be accurate.	Check balance maintenance, qualify data.
		Method Blank	1 per batch of 20 samples	Less than reported detection limits	Reanalyze all samples greater than MDL but less than 10x blank concentration.
		Duplicates	1 per batch of 20 samples	RPD ≤ 20%	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALY METHO	YTICAL OD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	
					THE WATER CHIEFLA	CORRECTIVE ACTION
SW846	7060A 7421	Total Arsenic Total Lead	FIELD OC:		•	
	7471A 7740 7841 7041	Total Mercury Total Selenium Total Thallium Total Antimony	Duplicate	l for every 10 field samples collected	Above 10x detection limit, % RPD must be less than current control limits: Aqueous samples - RPD < 20% Non-aqueous samples - RPD < 35%	Review lab QC data to determine if they are in control. I not in control, flag data. Use data to evaluate whether proper collection procedures were followed. If not, determine further corrective action.
			Rinsate (c)	1 for every 10 field samples collected	Less than reported detection limits	Qualify data.
			LABORATORY OC:			
			ICV/CCV	ICV - prior to analysis CCV - after every 10 samples or end of analytical batch, whichever is more frequent.*	Initial calibration each day - 3 stds plus blank. Verification measured value within 10% of true value using 1 blank & 1 mid-range. If samples >10, CCV within 20% of true.	Terminate analysis, solve problem, recalibrate and reanalyze samples analyzed since last good CCV.
		ICB/CCB	ICB - after initial calibration verification CCB - after every 10 samples or end of analytical batch, whichever is more frequent.*	Absolute value < PQL	Terminate analysis, solve problem, recalibrate and reanalyze samples analyzed since last good CCB.	
			Method Blank	l per batch of samples, minimum of l per 20 samples	Absolute value < PQL*	Redigest and reanalyze all samples less than 10X the PQL
			Matrix Spike	1 per batch of samples, minimum of 1 per 20 samples	75-125% recovery (unless sample conc. is greater than 4x spike concentration). Spike 5X above background at minimum.	Determine cause, then respike.* If uncorrectable, correct for bias if recovery is <80%.
		Matrix Spike Duplicate	I per batch of samples, minimum of I per 20 samples	20% RPD for samples greater than 5x PQL; if < 5x PQL, absolute difference between samples must be < PQL; no criteria if < PQL; (for Hg, RPD = 25% for aqueous samples and 35% for solid samples).*	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.	
			LCS	I per batch of samples, minimum of I per 20 samples.	75-125% recovery: waters Manufacturer's Limits: soil/sed.*	Rerun. If still out of control, solve problem and reanalyze batch.
		Serial Dilution	I per batch of samples, minimum 1 per 20 samples (for ICP only)	Diluted values must be < 10% of the original value	Perform recovery test (see below)	
		Recovery Test	When results from dilution test fail. Test is run on the failed sample.	85-115% recovery	Run Method of Standard Additions (MSA)	
			MSA	For each analyte where matrix interference is suspected or when recovery test fails	Slope within 20% of standard curve.	Qualify all associated data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE OF THE STATE OF THE	
				ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW846 6010A Inductively	Total Metals	FIELD QC:	•		
Coupled Argon Plasma (ICAP)		Duplicate	I for every 10 field samples collected	Aqueous samples - RPD < 30%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed. If not,
		Rinsate (c)	1 for every 10 field samples collected	No more than 4 target compounds, each with a concentration exceeding 3 times the method detection limit can be present.	determine further corrective action. Qualify data.
		LABORATORY OC:			
		Initial (ICV) and Continuing (CCV) Calibration Verification	ICV - prior to sample analysis and reanalysis of high standard. CCV - after every 10 samples and end of analytical batch	ICV - 4 pt. calibration (3 STDs and a blank); high standard within 5% of true value. CCV - midpoint range standard within 10% of true value.	Terminate analysis, solve problem, recalibrate and reanalyze samples analyzed since last good CCV.
		High Mixed Calibration Standard	Before begining of sample run	Agree within 10% of expected value	Follow recommendation of inst. manufacturer
		Continuing (CCB) Calibration Blank	CCB - after every 10 samples and end of analytical batch	Agree within ±3σ of mean blank value	Terminate analysis, solve problem, recalibrate and
			Method Blank	1 per batch of samples, minimum 1 per 20 samples	Absolute value s PQL*
		Serial Dilution	First encounter of new or unusual matrix	1:4 dilution agree within ±10% of original determination.	Flag as chemical or physical interference.
		Matrix Spike	1 per batch of samples, minimum 1 per 20 samples	75-125% recover (unless sample is greater than 4x spike concentration). Minimum 10X detection limit.	Determine cause (if possible), correct, and respike. If cause cannot be determined, flag.
		Duplicate	I per batch of samples, minimum I per 20 samples	20% RPD for samples greater than 5x PQL; if 5x PQL absolute difference between samples must be < PQL; no criteria if < PQL*	Determine cause (if possible), correct, and respike. If cause cannot be determined, flag
		Interference Check	Beginning and end of run or per 8 hour shift	80-120% recovery	Terminate analysis, solve problem, recalibrate and reanalyze samples analyzed since last good ICS.

ANALYTICAL METHOD (a.b)	PARAMETER	QUALITY CONTROL			
(4,5)	THOUNETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW846 9012	Cyanide	FIELD OC:			
		Duplicate	1 for every 10 field samples	Above 10x detection limit, % RPD must be less than current control limits: Aqueous samples - RPD < 20% Non-aqueous samples - RPD < 35%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed. If not,
		Rinsate (c)	1 for every 10 field samples	Less than reported detection limits	determine further corrective action.
		LABORATORY OC:	,	Less than reported detection limits	Qualify data.
		ICV/CCV	ICV - prior to sample analysis CCV - after every 10 samples and end of analytical batch	5 pt. calibration; Measured value within 15% of true value. Coefficient of correlation > 0.995	Terminate analysis, solve problem, recalibrate and reanalyze samples analyzed since last good CCV.
		ICB/CCB	ICB - after initial calibration verification CCB - after every 10 samples and end of analytical batch	Absolute value ≤ MDL	Terminate analysis, solve problem, recalibrate and reanalyze samples analyzed since last good CCB.
		Prep Blank	1 per batch of samples, minimum 1 per 20 samples	Absolute value ≤ MDL	Redigest and reanalyze all samples greater than the MDL but less than 10x the blank concentration.
		Spike	1 per batch of samples, minimum 1 per 20 samples	75-125% recovery (unless sample is greater than 4x spike concentration).	Perform a post-digestion spike and qualify data appropriately.
		Duplicate .	I per batch of samples, minimum I per 20 samples	20% RPD for samples greater than 5x MDL; if < 5x MDL, absolute difference between samples must be < MDL; no criteria if < MDL	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
		LCS	1 per batch of samples, minimum 1 per 20 samples	80-120% recovery: waters Manufacturer's Limits: soil/sed.	Rerun. If still out of control, solve problem and reanalyze batch. (Not applicable to mercury according to EPA Region II guidelines)

ANALYTICAL		QUALITY CONTROL			
METHOD (a,b)	PARAMETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
			· · · · · · · · · · · · · · · · · · ·		CORRECTIVE ACTION
EPA 130.2	Hardness	FIELD QC:			
		Duplicates	1 for every 10 field samples collected	% RPD ≤ 25%	Review lab QC data to determine if they are in control. If not, qualify data. Use data to evaluate proper collection procedures were followed. If not, determine further
		Rinsates (c) LABORATORY QC:	1 for every 10 field samples collected	Less than reported detection limits	Corrective action. Qualify data.
		Standardization of standards and reagents	At the beginning and end of the sample run	Standards must be ± 1.0% of the true value.	Check standards and reagents and prepare new if necessary.
		Method Blank Duplicate Matrix Spike	1 per batch of 20 samples 1 per batch of 20 samples 1 per batch of 20 samples	Less than reported detection limits % RPD ≤ 20% See laboratory control limits	Reanalyze all samples greater than MDL but less than 10x blank concentration. Qualify data.
				-	Reanalyze, qualify data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY		
			TREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW846 9310	Gross Alpha and Gross Beta	FIELD OC: Duplicate	I for every 10 field samples	Above 10x detection limit, % RPD must be less than current control limits	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether
÷		Rinsate (c) LABORATORY OC:	1 for every 10 field samples	Less than reported detection limits	proper collection procedures were followed. If not, determine further corrective action. Qualify data.
		Method Blank	1 for every 20 samples or extraction batch	Less than reporting detection limit	Document and report to client.
		Duplicate LCS	1 for every 20 samples or extraction batch 1 for every 20 samples or extraction batch	Above 10x detection limit, % RPD must be less than current control limits Within current control limits established by the laboratory	Review lab QC data to determine if they are in control. If not in control, flag data. Rerun samples. If still out of control, reanalyze samples. Qualify data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL			
(u,0)	TARAWETER	СНЕСК	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW846 7196	Chromium, Hexavalent	FIELD OC: Duplicate	I for every 10 field samples	Above 10x detection limit, % RPD must be less than current control limits: Aqueous samples - RPD < 30%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether
		Rinsate (c) <u>LABORATORY OC</u> :	1 for every 10 field samples	Non-aqueous samples - RPD < 40% Less than reported detection limits	proper collection procedures were followed. If not, determine further corrective action. Qualify data.
		Method Blank	1 for every 10 samples or extraction batch	Less than reporting detection limit	Document and report to client.
		MS/MSD Continuing Calibration	1 for every 10 samples 1 for every 15 samples	See Appendix M for control limits Within current control limits	Document and report to client. Terminate analysis, solve problem. Recalibrate and reanalyze samples analyzed force less than the control of
		LCS/LCSD Duplicate	1 for every 10 samples 1 for every 10 samples	80-120% Recovery, RPD ≤ 20% Above 10x detection limit, % RPD must be less than the control limits: Aqueous samples - RPD ≤ 30% Non-aqueous samples - RPD ≤ 40%	reanalyze samples analyzed from last good continuing calibration. Terminate analysis, solve problem. Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL		QUALITY CONTROL			
METHOD (a,b)	PARAMETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	COPPEGENT
				The Country of the Co	CORRECTIVE ACTION
NING P.O.			•		
NYS-DOH: APC 44	Glycols	FIELD QC:			
		Duplicate	l for every 10 field samples collected	% RPD must be ≤ 25%	Review lab QC data to determine if they are in control. If not, qualify data. Use data to evaluate proper collection
		Rinsates (c)	1 for ayon, 10 Cald		procedures were followed. If not, determine further corrective action.
			1 for every 10 field samples collected	Less than reported method detection limits	Qualify data.
		LABORATORY OC:			
		Initial Calibration	At the beginning of the run	3 pt. calibration; Coefficient of correlation must be ≤ 0.995	Terminate analysis, solve problem. Recalibrate and reanalyze samples.
		Continuing Calibration	After every 10 samples	Results must be ± 10% of the true value	
		Preparation Blank	I man harabase Company		Terminate analysis, solve problem. Recalibrate and reanalyze samples analyzed from last good continuing calibration.
			1 per batch of samples, minimum 1 per 20 samples	Value must be less than reported detection limit.	Reanalyze all samples greater than MDL but less than 10x blank concentration.
		MS/MSD	1 per batch of samples, minimum 1 per 20 samples	Recovery must be 75-125%	
			per 20 sampies	% RPD must be ≤ 20%	Rerun. If still out of control, reanalyze. Qualify data.
		Duplicate	I per batch of samples, minimum 1 per 20 samples	Recovery must be 75-125% % RPD must be ≤ 20%	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL		QUALITY CONTROL			
METHOD (a,b)	PARAMETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	
				HOOSI WINCE CIGITARY	CORRECTIVE ACTION
			•		
SW846 8140	Organo- phosphorus	FIELD QC:			
	Pesticides	Duplicate	1 for every 10 field samples collected	Water Samples - RPD < 20% Soil Samples - RPD < 35%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed.
		Rinsate (c)	1 for every 10 field samples collected	Less than reported detection limits	Qualify data.
		LABORATORY QC:	•	·	
		Initial Calibration	Prior to analysis and when continuing calibration fails criteria	5 pt. calibration; the average response factor can be used if %RSD is ≤ 20% or use a calibration curve	Recalibrate instrument.
		Continuing Calibration	Daily	CCCs response factor ≤ 15% difference from the midpoint standard	Rerun continuing calibration. If still out of control,
		Method Blank	One per extraction batch	Compounds must be below respective PQLs	recalibrate instrument.
		Surrogate Spike	All blanks, standards, QC samples, field samples	1,3-dimethyl-2-nitrobenzene Waters: 60-120 Soils: 50-120	Reextract batch. Reextract that sample.
		MS/MSD	l per every 20 samples	See method for control limits.	Determine cause (if possible), correct and reanalyze. If
		LCS	l per every 20 samples	See metihod for control limits.	cause cannot be determined, flag data. Reextract batch.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	EDEOLIENOV		
			FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW846 8150	Herbicides	FIELD QC:			
		Duplicate	1 for every 10 field samples collected	Water Samples - RPD < 20% Soil Samples - RPD < 35%	Review lab QC data to determine if they are in control. I not in control, flag data. Use data to evaluate whether proper collection procedures were followed.
		Rinsate (c)	1 for every 10 field samples collected	Less than reported detection limits	Qualify data.
		LABORATORY QC:			
		Initial Calibration	Prior to analysis and when continuing calibration fails criteria	5 pt. calibration; the average response factor can be used if %RSD is ≤ 20% or use a calibration curve	Recalibrate instrument.
		Continuing Calibration	Daily	CCCs response factor ≤ 15% difference from the midpoint standard	Rerun continuing calibration. If still out of control,
		Method Blank	One per extraction batch	Compounds must be below respective PQLs	recalibrate instrument.
		Surrogate Spike	All blanks, standards, QC samples, field samples	2,4-dichlorophenyl acetic acid (DCAA) Waters: 60-120 Soils: 50-120	Reextract batch. Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
		MS/MSD	1 per every 20 samples	See laboratorty limits.	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
		LCS	I per every 20 samples	See laboraoty limits.	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL			
	THE BUILTER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW846 8080A	Pesticides/PCBs	FIELD OC:			
		Duplicate	I for every 10 field samples collected 5% (wipe samples)	Above 10x detection limit, % RPD must be less than current control limits: Aqueous samples - RPD < 30% Non-aqueous samples - RPD < 35%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed. If not, determine further corrective action.
		Rinsate (c)	1 for every 10 field samples collected	Less than reported detection limit	Qualify data.
		Field Blanks	(wipe samples only) 2 from each category	Less than reported detection limit	Qualify data.
		LABORATORY QC:			
		Initial Calibration	Prior to analysis and when continuing calibration fails criteria	5 pt. calibration; the average response factor can be used if %RSD is ≤ 20% or use a calibration curve	Recalibrate instrument.
		Continuing Calibration	Daily and after every 10 samples	Response factor ≤ 15% difference from midpoint standard	Rerun continuing calibration. If still out of control, recalibrate instrument.
		Endrin and DDT Breakdown	Each initial calibration	Must not exceed 20%	Reanalyze breakdown standard. If still out of control along
		Combined Endrin and	Each initial calibration		injection port, change septae, replace first few inches of packing in column.
		DDT Breakdown	Such minut Cambration	Must not exceed 30%	Reanalyze breakdown standard. If still out of control, clean injection port, change septae, replace first few inches of
		Method Blank	1 for every 20 samples or extraction batch	Less than PQL	Reanalyze blank. If second blank exceeds critoric along
	S	Surrogate Recovery	Every sample	See Table 3 and 4 in method	analytical system. Quality the data.
		MS/MSD	1.0	and 4 in memod	Rerun sample. If still out of control, reextract, reanalyze, qualify data.
		LCS	I for every 20 samples	See Table 3 and 4 in method	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
		LCS	1 for every 20 samples	See Table 3 and 4 in method	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL			
	THUBINETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CODDECTIVE ACTION
			:		CORRECTIVE ACTION
SW846 8260A	Volatile Organic Compounds	FIELD OC:			
	- ompounds	Trip Blank	I for each batch of samples	N	
		Post c	shipped to laboratory	No more than 4 target compounds, each with a concentration exceeding 3 times the method detection limit can be present.	Review lab QC data to determine if there is a laboratory problem. If not, and same compounds are found in field samples at similar concentrations, resample entire batch.
		Duplicate	I for every 10 field samples	Above 10x detection limit, % RPD must be less	
		D' (A)	collected	than current control limits: Aqueous samples - RPD < 30% Non-aqueous samples - RPD < 40%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed. If not, determine further corrective action.
		Rinsate (c)	I for every 10 field samples collected	Less than reported PQL	Qualify data.
		LABORATORY QC:			
		Harware tune with BFB	At begining of analytical sequence and every 12 hours of operation thereafter	lon abundance criteria; see method	Tune instrument; repeat.
:		Initial Calibration	Prior to analysis and when continuing calibration fails criteria	5 pt. calibration; SPCCs ≥0.300; 1,1,2,2-TCA ≥ 0.200; bromoform ≥0.100. RSD <30% for RF for CCCs.	Recalibrate instrument.
		Continuing Calibration	Every 12 hours of operation	SPCCs ≥0.300, except 1,1,2,2-TCA ≥0.200 & bromoform ≥0.100. RSD <25% FOR avg RF for CCCs.	Rerun continuing calibration. If still out of control, recalibrate instrument.
		Method Blank	l for every 20 samples or every day	Less than PQL	Call AE chemist for further evaluation or,
·		Surrogate Recovery	Every sample	See Method	Reanalyze blank. If contamination still exists, qualify all associated data. 1. Check for errors during analysis. If found, recalculate. 2. Check instrument performance. Correct problem and reanalyze.
		MS/MSD	I for every 20 samples	See Method	3. If no problem found, re-extract and re-analyze sample. If problem persists, flag data. Analyze LCS. If more than 30% of either MS or MSD is outside telegrapes.
		LCS	1 for every 20 samples	See Method	above. Call AE chemist for guidance. Reanalyze LCS. If out, correct problem. If problem cannot be corrected, reject data.

ANALYTICAL		QUALITY CONTROL			
METHOD (a,b)	PARAMETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
			•		CONTROL ACTION
SW846 8100	Polynuclear Aromatic	■FIELD OC:			
	Hydrocarbons	Duplicate	1 for every 10 field samples collected	Water Samples - RPD < 20% Soil Samples - RPD < 30%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether
		Rinsate (c)	I for every 10 field samples collected	Less than reported detection limits	proper collection procedures were followed. Qualify data.
		LABORATORY OC:		•	
		Initial Calibration	Prior to analysis and when continuing calibration fails criteria	5 pt. calibration; % RSD must be < 20 % for all analytes	Recalibrate instrument.
		Continuing Calibration	Daily .	Response factor must be < 15% from average of initial calibration	Rerun continuing calibration. If still out of control, recalibrate instrument.
		Method Blank	Daily	Compounds must be below respective detection limits	Step 1: Reanalyze Step 2: If second blank exceeds criteria, clean the analytical system. Step 3: Document the corrective action taken and qualify.
		Surrogate Spike	All blanks, standards, QC samples, field samples	See Appendix M for current control limits	Step 1: Reanalyze Step 2: If recovery still outside control limits, re-extract
		MS/MSD	1 per every 20 samples	See Appendix M for current control limits	Rerun samples. If still out of control, qualify data.

ANALYTICAL METHOD (a,b)	DAD ALGEBER	QUALITY CONTROL			
METHOD (a,0)	PARAMETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
					CORRECTIVE ACTION
SW846 8040	Phenols	FIELD OC:			
		Duplicate	I for every 10 field samples collected	Water Samples - RPD < 20% Soil Samples - RPD < 30%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether
		Rinsate (c)	1 for every 10 field samples collected	Less than reported detection limits	proper collection procedures were followed. Qualify data.
		LABORATORY QC:			
		Initial Calibration	Prior to analysis and when continuing calibration fails criteria	5 pt. calibration, % RSD must be < 20% for all analytes	Recalibrate instrument.
		Continuing Calibration	Each day of operation	Response factor must be < 15% from average of initial calibration	Rerun continuing calibration. If still out of control, recalibrate instrument.
		Method Blank	Each day of operation	Compounds must be below respective detection limits	Step 1: Reanalyze. Step 2: If second blank exceeds criteria, clean the analytical system.
		Surrogate Spike			Step 3: Document the corrective action taken and qualify all associated data.
			All blanks, standards, QC samples, field samples	See Appendix M for current control limits	Step 1: Reanalyze. Step 2: If recovery still outside control limits, re-extract and reanalyze.
		MS/MSD	I per every 20 samples	See Appendix M for current control limits	Rerun samples. If still out of control, qualify data.

ANALVEICAL					
ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY		
			TAEQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW846 8270B GC/MS	Base/Neutral/ Acid Extractable	FIELD QC:			
	Organics	Duplicate	I for every 10 field samples	Above 10x detection limit, % RPD must be less	D : 1100
			collected	than current control limits: Aqueous samples - RPD < 20% Non-aqueous samples - RPD < 35%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed. If not, determine further corrective action.
		Rinsate (c)	1 for every 10 field samples collected	Less than reporting limit	Qualify data.
	•	LABORATORY QC:			
		Sensitivity Check with DFTPP	At beginning of each analytical run & beginning of each 12 hr period	Ion abundance criteria; see method	Tune instrument; repeat. If cannot be corrected, reject data.
		Mass Calibration*	Every 24 hours & at beginning of each analytical sequence	See Method SW846	Tune instrument; repeat.
		Initial Calibration	Before analysis and when continuing calibration fails criteria	5 pt. calibration; SPCCs exceed 0.050; CCCs response factor deviates < 30% from average	Correct problem (inj. liner,column). Recalibrate instrument. Reject if problem not solved.
		Continuing Calibration	Every 12 hours of operation	SPCCs ≥0.050; CCCs ≤ 30% from standard concentration. RT of IS ≤ 30 sec over 12 hours & EICP area changes within -50% to +100%.	Correct problem and rerun continuing calibration; if still out of control, recalibrate instrument. Reanalyze samples. Reject if problem not solved.
		Method Blank	I for every 20 samples or extraction batch	Less than PQL. Phthalate esters less than 5x the reporting limit.*	Reanalyze blank, then reextract if necessary if holding time and sample volume allows. Qualify all associated data. If HT and sample volume precludes reanalysis, contact AE
		Surrogate Recovery	Every sample	0. 14.4.5	chemist.
			zvery sumple	See Method. Recovery for at least 2 of 3 acid surrogates and at least 2 of 3 BN surrogates must be within tolerance.	Call A-E chemist, or 1. Check calculations & instrument performance 2. If problem found, correct and recalculate and/or reanalyze extract. 3. If problem for found, re-extract & re-analyze sample.
		MS/MSD	1 for every 20 samples		4. If still out, flag data as estimated.
		LCS	·	See Method	Run LCS. If LCS not within Table 6, see below. If LCS is good, and assignable cause is found for poor MS/MSD, correct and redo MS/MSD (see text).* Otherwise, flag data.
	•	LCS	1 for every 20 samples or extraction batch	See Method	Reextract batch if MS/MSD also out of control.*

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA 120.1	Specific Conductance	INSTRUMENT OC: Calibration Calibration Stability SAMPLE OC: Duplicate Rinsate (c)	Prior to trip At beginning and end of day 1 per day 1 per day	± 25 umhos/cm ± 25 umhos/cm ± 50 units ≤ detection limit	 Check system as per manufacturer's instructions. Check standard. Replace instrument. Check standard. Check system as per manufacturer's instructions. Replace instrument. Analyze 3rd aliquot of sample. Flag Data.
					i lag Data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA 150.1	рН	INSTRUMENT OC:	l per day at two levels	± 0.1 units	Check system as per manufacturer's instructions.
		Calibration Stability SAMPLE OC:	1 per hour at two levels	± 0.2 units	2. Check standard. 3. Replace instrument. 1. Check standard. 2. Check system. 3. Recalibrate.
		Duplicate Rinsate (c)	l per day	± 0.5 units ≤ detection limit	Analyze 3rd aliquot of sample Flag data. Flag data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA 180.1	Turbidity	INSTRUMENT QC:	l mor ilou		
		Calibration Stability	l per day	± 25 NTU ± 25 NTU	Check system as per manufacturer's instructions. Check standards. Replace Instrument. Check standards. Check system as per manufacturer's instructions.
		SAMPLE QC:			Recalibrate.
		Duplicate	l per day	± 50 NTU	Analyze 3rd aliquot of sample.
		Rinsate (c)	l per day	≤ detection limit	2. Flag data. Flag data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA 9060	TOC	FIELD OC:			CORRECTIVE ACTION
		Duplicate Rinsate (c) LABORATORY OC:	Every 20 samples Every 20 samples	RPD: <35% ≤ detection limit	Flag data.
		Calibration	At start of analysis	see method	 Check standard solution. Check system as per manufacturer's instructions. Perform appropriate instrument maintenance.
		Calibration Stability Method Blank	At end of analysis	± 1 mg/L	 Check standard solution. Check system. Recalibrate and reanalyze samples.
	Duplicate	I per batch of samples analyzed together, minimum I per 20 samples	≤ 3x detection limit 1. Check system. 2. Check blank. 3. Flag data.	Check system. Check blank.	
		Duplicate Minimum 1 per 20 field samp	amples	RPD: <30%	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA 600 4-83-043	Asbestos	FIELD OC: QC Sample LABORATORY OC: Duplicate	1 for every 20 field samples 1 for every 20 samples	No significant difference (detect vs. non-detect, different type of asbestos) from reference sample 2 analysts identify sample with no significant difference	Reject sample and reinspect/retest corresponding area. Review by Senior Engineer and both analysts.

ANALYTICAL	_	QUALITY CONTROL			
METHOD (a,b)	PARAMETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORDECTIVE
					CORRECTIVE ACTION
EDA OSCO			•		
EPA 906.0	Tritium	FIELD QC:			
		Duplicate	1 for every 10 field samples	Above 10x detection limit, % RPD must be less	.
			·	than current control limits	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether
•				Less than reported detection limits	proper collection procedures were followed. If not, determine further corrective action.
	•	Rinsate (c)	1 for every 10 field samples		Qualify data.
		LABORATORY OC:			Quality data.
		Method Blank	1 for every 20 samples or extraction batch	Less than reporting detection limit	Document and report to client.
		Duplicate	1 for every 20 samples or	Above 10v detection 1' is as pro-	
		extraction batch	Above 10x detection limit, % RPD must be less than current control limits established by the laboratory	Review lab QC data to determine if they are in control. If not in control, flag data.	
•		LCS	I for every 20 samples or extraction batch	Within current control limits established by the laboratory	Rerun samples. If still out of control, reanalyze samples. Qualify data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL			
		СНЕСК	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
					CONTROL ACTION
ANL-Eichrom	Strontium-90	FIELD OC:			
		Duplicate	1 for every 10 field samples	Above 10x detection limit, % RPD must be less than current control limits	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether
		Rinsate (c) <u>LABORATORY OC</u> :	I for every 10 field samples	Less than reported detection limits	proper collection procedures were followed. If not, determine further corrective action. Qualify data.
		Method Blank	1 for every 20 samples or extraction batch	Less than reporting detection limit	Document and report to client.
		Duplicate	1 for every 20 samples or extraction batch	Above 10x detection limit, % RPD must be less than current control limits established by the laboratory	Review lab QC data to determine if they are in control. If not in control, flag data.
		LCS	1 for every 20 samples or extraction batch	Within current control limits established by the laboratory	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FDFOUR		
			FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA 903.1	Radium-226	FIELD QC:			
		Duplicate	I for every 10 field samples	Above 10x detection limit, % RPD must be less than current control limits	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether
		Rinsate (c) <u>LABORATORY QC</u> :	1 for every 10 field samples	Less than reported detection limits	proper collection procedures were followed. If not, determine further corrective action. Qualify data.
		Method Blank	1 for every 20 samples or extraction batch	Less than reporting detection limit	Document and report to client.
		Duplicate	1 for every 20 samples or extraction batch	Above 10x detection limit, % RPD must be less than current control limits established by the laboratory	Review lab QC data to determine if they are in control. If not in control, flag data.
		LCS	1 for every 20 samples or extraction batch	Within current control limits established by the laboratory	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL			
	THE BYIETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
					CORRECTIVE ACTION
EPA 901.1	Gamma-emitting Radionuclides	FIELD OC:			
	Radionachues	Duplicate	1 for every 10 field samples	Above 10x detection limit, % RPD must be less than current control limits	Review lab QC data to determine if they are in control. If
		Rinsate (c)	1 for every 10 field samples		not in control, flag data. Use data to evaluate whether proper collection procedures were followed. If not, determine further corrective action.
		LABORATORY OC:		Less than reported detection limits	Qualify data
		Method Blank	1 for every 20 samples or extraction batch	Less than reporting detection limit	Document and report to client
		Duplicate	1 for every 20 samples or extraction batch	Above 10x detection limit, % RPD must be less than current control limits established by the	Review lab QC data to determine if they are in control. If not in control, flag data.
		LCS	1 for every 20 samples or extraction batch	Within current control limits established by the laboratory	Determine cause (if possible) correct and registers. It
•			•		cause cannot be determined, flag data.

ANALYTICAL METHOD (a,b) PARAMET	QUALITY CONTROL FER CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
				CONDUCTIVE ACTION
EPA 904.0 Radium-228	FIELD QC:			·
	Duplicate	1 for every 10 field samples	Above 10x detection limit, % RPD must be less than current control limits	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed. If not, determine further corrective action.
•	Rinsate (c)	1 for every 10 field samples	Less than reported detection limits	Qualify data.
	LABORATORY QC:			
	Method Blank	1 for every 20 samples or extraction batch	Less than reporting detection limit	Document and report to client.
	Duplicate ,	1 for every 20 samples or extraction batch	Above 10x detection limit, % RPD must be less than current control limits established by the laboratory	Review lab QC data to determine if they are in control. If not in control, flag data.
	LCS	1 for every 20 samples or extraction batch	Within current control limits established by the laboratory	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
			•	

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA 908.0	Total Uranium	FIELD OC: Duplicate	1 for every 10 field samples	Above 10x detection limit, % RPD must be less than current control limits	Review lab QC data to determine if they are in control. If
		Rinsate (c) LABORATORY OC:	1 for every 10 field samples	Less than reported detection limits	not in control, flag data. Use data to evaluate whether proper collection procedures were followed. If not, determine further corrective action. Qualify data.
	÷	Method Blank	1 for every 20 samples or extraction batch	Less than reporting detection limit	Document and report to client.
		Duplicate	1 for every 20 samples or extraction batch	Above 10x detection limit, % RPD must be less than current control limits established by the laboratory	Review lab QC data to determine if they are in control. If not in control, flag data.
		LCS	1 for every 20 samples or extraction batch	Within current control limits established by the laboratory	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
		LABORATORY OC: Method Blank Duplicate	1 for every 20 samples or extraction batch 1 for every 20 samples or extraction batch 1 for every 20 samples or	Less than reporting detection limit Above 10x detection limit, % RPD must be less than current control limits established by the laboratory Within current control limits established by the	proper collection procedures were followed. If not, determine further corrective action. Qualify data. Document and report to client. Review lab QC data to determine if they are in control in control, flag data. Determine cause (if possible), correct and reanalyze.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA 350.2	Ammonia Nitrogen	FIELD OC: Duplicate	I for every 10 field samples	The % RPD must be < 20	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether
		Rinsate (c) <u>LABORATORY QC:</u>	l for every 10 field samples	Less than reported detection limits	proper collection procedures were followed. If not, determine further corrective action. Qualify data.
		Method Blank	1 for every 20 samples or extraction batch	Less than reported detection limit	Document and report to client.
		Lab Duplicate	1 for every 20 samples	RPD must be < 20	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
		Calibration	Prior to analytical run	Coefficient of correlation must be > 0.995	Check standard solutions. Qualify data.
		Continuing Calibration	1 per 10 sample and at the end of the run	± 0.10 mg/L	Check standard solutions. Recalibrate. Reanalyze affected samples. Qualify data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA 340.1	Total Fluoride	FIELD QC:			CONGRETATE ACTION
		Duplicate Rinsate (c) <u>LABORATORY QC:</u>	1 for every 10 field samples One for every 10 field samples	RPD: <20% ≤ detection limit	Flag data Flag data
		Calibration	At start of analysis	Coefficient of correlation must be > 0.995	Check standard solution Reprepare standard
		Calibration Stability	Every 10 samples and at end of analysis	± 1 mg/L	Check standard solution Recalibrate and reanalyze samples
		Method Blank	I per batch of samples analyzed together, minimum I per 20 field samples	≤ detection limit	1) Check blank 2) Flag data
		Duplicate	Minimum 1 per 20 field samples	RPD: <20%	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA 425.1	Linear Alkyl Benzene Sulfonate	FIELD OC: Duplicate Rinsate (c) LABORATORY OC:	See Table 5-1 See Table 5-1	RPD: <20% ≤ detection limit	Flag data Flag data
		Calibration	At start of analysis	Coefficient of correlation must be > 0.995	Check standard solution Reprepare standard
		Calibration Stability	Every 10 samples and at end of analysis	± 1 mg/L	Check standard solution Recalibrate and reanalyze samples
		Method Blank	1 per batch of samples analyzed together, minimum 1 per 20 field samples	≤ detection limit	Check blank Flag data
		Duplicate	Minimum 1 per 20 field samples	RPD: <20%	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
			·		CONTRACTION ACTION
EPA 300.0	Nitrate/Sulfate	FIELD QC:			
		Duplicate	See Table 5-1	RPD: <20%	Flag data
•		Rinsate (c)	See Table 5-1	≤ detection limit	Flag data
		LABORATORY OC:			
		Calibration	At start of analysis	Coefficient of correlation must be > 0.995	1) Check standard solution 2) Reprepare standard
		Calibration Stability	Every 10 samples and at end of analysis	± 1 mg/L	Check standard solution Recalibrate and reanalyze samples
•		Method Blank	1 per batch of samples analyzed together, minimum 1 per 20 field samples	≤ detection limit	I) Check blank 2) Flag data
		Duplicate	Minimum 1 per 20 field samples	RPD: <20%	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	
				TOOL THEE CHIENA	CORRECTIVE ACTION
EPA 377.1	Sulfite	FIELD OC:			
		Duplicate	See Table 5-1	RPD: 20%	Flag data
		Rinsate (c)	See Table 5-1	≤ detection limit	Flag data
		LABORATORY OC:			· ing onto
·		Calibration	At start of analysis	Coefficient of correlation must be > 0.995	Check standard solution Reprepare standard
		Calibration Stability	Every 10 samples and at end of analysis	± 1 mg/L	Check standard solution Recalibrate and reanalyze samples
		Method Blank	I per batch of samples analyzed together, minimum I per 20 field samples	≤ detection limit	1) Check blank 2) Flag data
		Duplicate	Minimum 1 per 20 field samples	RPD: <20%	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA 330.5	Total Residual Chlorine	FIELD QC:	•	,	
		Calibration	At start of analysis	Coefficient of correlation must be > 0.995	Check standard solution Reprepare standard
		Method Blank	1 per batch of samples analyzed together, minimum 1 per 20 field samples	< detection limit	Check blank Flag data
	,	Duplicate .	I per batch of samples analyzed together, minimum I per 20 field samples	RPD: < 20%	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
		Rinsate (c)	I per batch of samples analyzed together, minimum 1 per 20 field samples	s detection limit	Flag data

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
		· · · · · · · · · · · · · · · · · · ·		MODEL WATER CHATERIA	CORRECTIVE ACTION
EPA 524.2	Purgeable Organic Compounds	FIELD QC:			
	- Compounds	Duplicate	I for every 10 field samples collected	Water Samples - RPD < 20% Soil Samples - RPD < 35%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed.
		Field Reagent Blank	l per cooler	Less than reported POL	Qualify data.
		Rinsate (c)	I for every 10 field samples collected	Less than reported PQL	Qualify data.
		LABORATORY OC:	٠.٠	÷	
		Initial Calibration	Prior to analysis and when continuing calibration criteria fail	4 pt. calibration; RSD of mean < 20% or 2nd order regression	Recalibrate instrument.
		Continuing Calibration	Every 8 hours of operation	70-130% of true value. Absolute areas of quantitation ions of the internal and surrogate	Rerun continuing calibration. If still out of control, take remedial action and recalibrate instrument.
		· · · · · · · · · · · · · · · · · · ·		standards must not have decreased by more than 30% from areas in the most recent continuing calibration check or by more than 50% from the areas in the initial calibration.	
		Laboratory Reagent Blank	Every 8 hours of operation	Less than reported PQL	Step 1: Reanalyze. Step 2: If second blank exceeds criteria, clean the analytical system. Step 3: Document the corrective action taken and qualify all associated data.
		QC Check Standard (External Source)	Quarterly .	%R = 80-120 RSD < 20%	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Reanalyze.
		Sensitivity Check with BFB	Every 8 hours of operation	Ion abundance criteria; see method	Tune instrument; repeat.
		Mass Calibration	Every 8 hours of operation	See method	Tune instrument; repeat.
		Laboratory Fortified Blank - Full Method Analyte List	1 per 20 or 1 per sample set	LFB: 80-120%R Except: 70-130%R on 1,2-dibromo-3-chloropropane Hexachlorobutadiene Naphthalene 1,2,3-Trichloropropane 2,2-Dichloropropane Advisory: 60-140%R on additional special compounds (nonroutine) by 524.2	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Reanalyze.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA 524.2 (Continued)	Purgeable Organic Compounds	Laboratory Fortified Blank Duplicate	Quarterly	RPD < 20%; %R same as LFB	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Reanalyze.
		Surrogate Spike MS/MSD	All blanks, standards, QC samples, field samples I per every 20 samples	See method for control limits See method for control limits	Step 1: Reanalyze Step 2: If recovery still outside control limits, qualify the data. Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL		QUALITY CONTROL			
METHOD (a,b)	PARAMETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CÓRRECTIVE ACTION
			: ·		
EPA 525.1	Organic Compounds	FIELD OC:			
		Duplicate	1 for every 10 field samples collected	Water Samples - RPD < 20%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed.
		Field Reagent Blank	1 per set of field samples	Less than PQL	Qualify data.
		Rinsate (c)	1 for every 10 field samples collected	Less than PQL	Qualify data.
		LABORATORY QC:			
		Initial Calibration	Prior to analysis and when continuing calibration criteria fail	6 pt. calibration; RSD of mean < 30% or use first-degree fit. Anthracene and phenanthrene should be separated by baseline. Benz[a]anthracene and chrysene should be separated by a valley whose height is less than 25% of the average peak height of these two compounds. The GC/MS/DS peak identification software should be able to recognize a GC peak in the appropriate retention time window for each of the compounds in calibration solution, and make correct tentative identifications. Examine a plot of the abundance of m/z 67 in the region of 1.05-1.3 of the retention time of endrin. This is the region of elution of endrin aldehyde, a product of the thermal isomerization of endrin. Confirm that the abundance of m/z 67 at the retention time of endrin aldehyde is <10% of the abundance of m/z 67 produced by endrin.	Recalibrate instrument. If the valley between benz[a]anthracene and chrysene exceeds 25%, the GC column requires maintenance. If more than 10% endrin aldehyde is observed, system maintenance is required to correct the problem.
		Continuing Calibration	Every 8 hours of operation	Response factors deviate < 30% from average of initial calibration. Absolute areas of quantitation ions of the internal and surrogate standards must not have decreased by more than 30% from areas in the most recent continuing calibration check or by more than 50% from the areas in the initial calibration.	Rerun continuing calibration. If still out of control, take remedial action and recalibrate instrument.
		Laboratory Reagent Blank	One per 20 samples or extraction batch	Less than PQL	Step 1: Reanalyze. Step 2: If second blank exceeds criteria, clean the analytical system. Step 3: Document the corrective action taken and qualify all associated data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	PRESIDENT		
(2,0)	THUGHTER		FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA 525.1 (Continued)	Organic Compounds	QC Check Standard (External Source)	Quarterly	%R = 70-130 RSD < 30%	Step 1: Reanalyze. Step 2: If still out of control, perform and document
		Sensitivity Check	Every 8 hours of operation	Ion abundance criteria; see method	remedial action. Step 3: Reanalyze.
		with DFTPP	or operation	ion abundance criteria, see method	Tune instrument; repeat.
		Mass Calibration	Every 8 hours of operation	See method	Tune instrument; repeat.
		Laboratory Fortified Blank - Full Method Analyte List	1 per 20 or 1 per sample set	See method	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action and qualify the data.
		Laboratory Fortified Blank Duplicate	Quarterly	See method	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action and qualify the data.
		Internal Standard	All blanks, QC samples, field samples	Recovery > 70% of standard response	Step 1: Reanalyze. Step 2: If recovery still outside control limits, qualify the data.
		Surrogate Spike	All blanks, standards, QC samples, field samples	See method for surrogate spiking compounds control limits	Step 1: Reanalyze. Step 2: If recovery still outside control limits, qualify the data.
		MS/MSD	l per every 20 samples	See method or laboratory controls	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL		QUALITY CONTROL			
METHOD (a,b)	PARAMETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
					CONNECTIVE ACTION
EDA SOS					
EPA 507	Nitrogen- and Phosphorus-	FIELD QC:			
	Containing Pesticides by GC/NPD	Duplicate	1 for every 10 field samples collected	Water Samples - RPD < 20% Soil Samples - RPD < 35%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed.
		Rinsate (c)	1 for every 10 field samples collected	Less than reported detection limits	Qualify data.
	•	Field Reagent Blank	l per cooler	Less than reported detection limits	Qualify data.
		LABORATORY OC:		,	Quanty data.
		Initial and Continuing Calibration	Prior to analysis and every 8 hours of operation	Single-point calibration (Sect. 9) within 20% of sample response	N/A
		Laboratory Reagent Blank	One per 20 samples or extraction batch	Less than PQL	Step 1: Reanalyze. Step 2: If second blank exceeds criteria, clean the analytical system. Step 3: Document the corrective action taken and qualify all associated data.
		QC Check Standard (External Source)	Quarterly	%R = 70-130	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action.
		Laboratory Fortified Blank with full method analytes list.	1 per 20 or 1 per sample set (all samples extracted within 24 hours)	See method or laboratory controls	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action and qualify data.
		Surrogate Spike	All blanks, standards, QC samples, field samples	%R = 70-130	Step 1: Reanalyze Step 2: If recovery still outside control limits for samples, qualify the data.
•		MS/MSD	1 per every 20 samples or 1 per set	See method or laboratory controls	Reanalyze MS/MSD. If still out of control, qualify data.

ANALYTICAL METHOD (a.k.)	D. D. L. Commission	QUALITY CONTROL			
METHOD (a,b)	PARAMETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
					COMMENTE ACTION
EPA 549	Diquat/Paraquat HPLC Method	FIELD OC:			
	TH EC Method	Duplicate	I for every 10 field samples collected	Water Samples - RPD < 20% Soil Samples - RPD < 35%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed.
		Field Reagent Blank	1 per cooler	Less than reported detection limit	Qualify data.
		Rinsate (c)	1 for every 10 field samples collected	Less than reported detection limits	Qualify data.
		LABORATORY OC:			
		Initial Calibration	Prior to analysis and when continuing calibration criteria fail	3 pt. external standard method calibration - use calibration curve or average RF if %RSD ≤ 20%; or use single-point calibration	Recalibrate instrument.
		Continuing Calibration	Every 8 hours of operation (two standards)	Response factors deviate ≤ 20% from midpoint standard.	Rerun continuing calibration. If still out of control, take remedial action and recalibrate instrument.
		Laboratory Reagent Blank	I for each set of extracted samples	Less than reported detection limit	Step 1: Reanalyze Step 2: If second blank exceeds criteria, clean the analytical system. Step 3: Document the corrective action taken and qualify all associated data.
		QC Check Standard (External Source)	Quarterly	%R = 80-120 RSD < 20%	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Reanalyze.
		Laboratory Fortified Blank	1 per 20 or 1 per sample set (all samples extracted within 24 hours)	%R Diquat = 58-124 Paraquat = 34-115	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Reanalyze.
		Laboratory Fortified Blank Duplicate	Quarterly	%R Diquat = 58-124 Paraquat = 34-115	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Reanalyze.
		MS/MSD	1 per every 20 samples	%R = 80-120 %RSD = 20%	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL		QUALITY CONTROL			
METHOD (a,b)	PARAMETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
					Totale IVE NOTION
EPA 515.1	Chlorinated Acids and Herbicides by GC/ECD	FIELD QC:	J.		
		Duplicate	1 for every 10 field samples collected	Water Samples - RPD < 20% Soil Samples - RPD < 35%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether
		Field Reagent Blank	1 per cooler	Less than reported detection limit	proper collection procedures were followed. Qualify data.
		Rinsate (c)	1 for every 10 field samples collected	Less than reported detection limits	Qualify data.
		LABORATORY OC:	•	•	
		Initial and Continuing Calibration	Prior to analysis and every 8 hours of operation	Single-point calibration (Section 9) within 20% of sample response	Correct problem. Reanalyze all affected samples.
	-	Laboratory Reagent Blank	One per 20 samples or extraction batch	Less than PQL	Step 1: Reanalyze. Step 2: If second blank exceeds criteria, clean the analytical system. Step 3: Document the corrective action taken and qualify all associated data.
		QC Check Standard (External Source)	Quarterly	%R = 80-120 RSD $\leq 20\%$	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
		Laboratory Fortified Blank - Full Method Analyte List	l per 20 samples or extraction batch	See method or laboratory controls	Determine cause (if possible), correct and reanalyze If
		Surrogate Spike	All blanks, standards, QC samples, field samples	See method or laboratory controls for surrogate spiking compounds control limits	cause cannot be determined, flag data. Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
		MS/MSD	1 per every 20 samples	See method or laboratory controls.	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL		OLIALITY CONTROL			
METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE
	· · · · · · · · · · · · · · · · · · ·			ACCEL PANCE CRITERIA	CORRECTIVE ACTION
EPA 504	EDB & DBCP by GC/ECD	FIELD OC:			
		Duplicate	I for every 10 field samples collected	Water Samples - RPD < 20% Soil Samples - RPD < 35%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed.
		Field Reagent Blank	l per cooler	Less than reported detection limit	Qualify data.
		Rinsate (c)	1 for every 10 field samples collected	Less than reported detection limits	Qualify data.
		LABORATORY QC:			
		Initial Calibration	Prior to analysis and when continuing calibration criteria fail	3-5 pt. or single-point calibration; RSD of mean < 20%	Recalibrate instrument.
		Continuing Calibration	Daily	60-140% of expected	Rerun continuing calibration. If still out of control, take remedial action and recalibrate instrument.
		Laboratory Reagent Blank	I for each batch of extracted samples	Less than PQL	Step 1: Reanalyze. Step 2: If second blank exceeds criteria, clean the analytical system. Step 3: Document the corrective action taken and qualify all associated data.
		QC Check Standard (External Source)	Quarterly	%R = 80-120 RSD < 20%	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
		Laboratory Fortified Blank	10% of samples	%R = 60-140 RSD < 40%	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
		MS/MSD	l per every 20 samples	See method or laboratory control %R = 60-140	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
					CORRECTIVE ACTION
EPA 548.1	Endothall by GC/FID	FIELD QC:			
		Duplicate	1 for every 10 field samples collected	Water Samples - RPD < 20%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed.
		Field Reagent Blank	1 per cooler	Less than reported detection limit	Qualify data.
		Rinsate (c)	1 for every 10 field samples collected	Less than reported detection limits	Qualify data.
		LABORATORY OC.			
		Initial Calibration	Prior to analysis and when continuing calibration criteria fail	4 pt. calibration; if RSD of RF ≤ 30%, use average RF or single-point calibration	Recalibrate instrument.
		Continuing Calibration	Every day	Response factors deviate ± 30% from initial calibration standard.	Rerun continuing calibration. If still out of control, take remedial action and recalibrate instrument.
		Laboratory Reagent Blank	One per 20 samples or extraction batch	Less than PQL	Step 1: Reanalyze. Step 2: If second blank exceeds criteria, clean the analytica system. Step 3: Document the corrective action taken and qualify all associated data.
		QC Check Standard (External Source)	Quarterly	%R = 80-120 RSD < 20%	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Reanalyze.
		Laboratory Fortified Blank	1 per sample set or 20 samples	%R = 57-107	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Reanalyze.
		MS/MSD	1 per every 20 samples	%R = 57-107	Reanalyze samples. If still out of control, qualify data.
		Internal Standard	All blanks, QC samples, field samples	%R = 70-130% Advisory	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Qualify data.

ANALYTICAL		QUALITY CONTROL			
METHOD (a,b)	PARAMETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
		•			CONNECTIVE ACTION
EPA 547	Glyphosate by HPLC	FIELD QC:			
		Duplicate	1 for every 10 field samples collected	Water Samples - RPD < 20% Soil Samples - RPD < 35%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed.
		Field Reagent Blank	l per cooler	Less than reported detection limit	Qualify data.
		Rinsate (c)	1 for every 10 field samples collected	Less than reported detection limits	Qualify data.
		LABORATORY OC:			
		Initial Calibration	Prior to analysis and when continuing calibration criteria fail	3 pt. calibration - use calibration curve or average RF if %RSD ≤ 20%; or use single-point calibration	Recalibrate instrument.
		Continuing Calibration	Each working day	Response factors deviate $\leq 20\%$ from midpoint standard.	Rerun continuing calibration. If still out of control, take remedial action and recalibrate instrument.
		Laboratory Reagent Blank	For each batch of samples or each day run	Less than reported detection limit	Step 1: Reanalyze. Step 2: If second blank exceeds criteria, clean the analytical system. Step 3: Document the corrective action taken and qualify all associated data.
		QC Check Standard (External Source)	Quarterly	%R = 80-120 RSD < 20%	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Reanalyze.
		Laboratory Fortified Blank	l per sample set, or all samples analyzed within 24 hours	%R = 70-130	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Reanalyze.
		MS/MSD	l per every 20 samples	%R = 80-120 RSD = 20%	Reanalyze samples. If still out of control, qualify data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
					CORRECTIVE ACTION
EPA 531.1	Carbamate Pesticides by HPLC	FIELD QC:			
		Duplicate	1 for every 10 field samples collected	Water Samples - RPD < 20% Soil Samples - RPD < 35%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed.
		Field Reagent Blank	1 per cooler	Less than reported detection limit	Qualify data.
		Rinsate (c)	1 for every 10 field samples collected	Less than reported detection limits	Qualify data.
		LABORATORY OC:			
		Initial Calibration	Prior to analysis and when continuing calibration criteria fail	3 pt. calibration - use calibration curve or average RF if %RSD ≤ 20%; or use single-point calibration	Recalibrate instrument.
		Continuing Calibration	Each day of operation	Response factors deviate ≤ 20% from midpoint standard.	Rerun continuing calibration. If still out of control, take remedial action and recalibrate instrument.
		Laboratory Reagent Blank	Each set of samples	Less than PQL	Step 1: Reanalyze. Step 2: If second blank exceeds criteria, clean the analytical system. Step 3: Document the corrective action taken and qualify all associated data.
		QC Check Standard (External Source)	Quarterly	%R = 80-120 RSD ≤ 20%	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Reanalyze.
		Laboratory Fortified Blank with full method analyte list	Every 8 hours of operation	%R = 30-170	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Reanalyze.
		MS/MSD	1 per every 20 samples	%R = 80-120 RSD = $\le 20\%$	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL		QUALITY CONTROL			
METHOD (a,b)	PARAMETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
					The Heriot
EPA 508	Pesticides by GC/ECD	FIELD OC:			
		Duplicate	I for every 10 field samples collected	Water Samples - RPD < 20% Soil Samples - RPD < 35%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed.
	,	Field Reagent Blank	l per cooler	Less than reported detection limit	Qualify data.
	•	Rinsate (c)	1 for every 10 field samples collected	Less than reported detection limits	Qualify data.
		LABORATORY OC:			
		Initial and Continuing Calibration	Every 12 hours of operation and prior to analysis	Single-point calibration (Section 9) within 20% of sample response	N/A
		Laboratory Reagent Blank	One per 20 samples or per batch	Less than PQL	Step 1: Reanalyze. Step 2: If second blank exceeds criteria, clean the analytical system. Step 3: Document the corrective action taken and qualify all associated data.
		QC Check Standard (External Source)	Quarterly	%R = 80-120 RSD < 20%	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action.
		Laboratory Fortified Blank with full method analytes list	1 per 20 or 1 per sample set	See method or laboratory controls	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action and qualify data.
		Surrogate Spike	All blanks, standards, QC samples, field samples	See method or laboratory controls.	Step 1: Reanalyze Step 2: If recovery still outside control limits, qualify the data.
		MS/MSD	1 per every 20 samples	See method or laboratory controls.	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
		Individual Endrin and DDT Breakdown	Daily	Must be < 20%	Perform and document remedial action. Qualify if corrective actions unsuccessful.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA 508 (Continued)	Pesticides by GC/ECD	Combined Endrin and DDT Breakdown	Daily 	Must be < 30%	Perform and document remedial action. Qualify if corrective actions unsuccessful

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL			
WETTOD (a,b)	PARAMETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA 8021	Volatile Organics by GC/FID/ELCD	FIELD OC:			
		Duplicate	1 for every 10 field samples collected	Water Samples - RPD < 20% Soil Samples - RPD < 35%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed.
		Field Reagent Blank	l per cooler	Less than reported detection limit	Qualify data.
		Rinsate (c)	I for every 10 field samples collected	Less than reported detection limits	Qualify data.
		LABORATORY QC:			
		Initial Calibration	Prior to analysis and when continuing calibration criteria fail	5 pt. calibration; RSD of mean < 20%	Recalibrate instrument.
		Continuing Calibration	Daily	Response factors deviate < 20% from average of initial calibration.	Rerun continuing calibration. If still out of control, take remedial action and recalibrate instrument.
		Laboratory Reagent Blank	Daily	Less than reported detection limit	Step 1: Reanalyze. Step 2: If second blank exceeds criteria, clean the analytical system. Step 3: Document the corrective action taken and qualify all associated data.
		QC Check Standard (External Source)	Quarterly	%R = 80-120 RSD < 20%	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Reanalyze.
		Laboratory Fortified Blank	I per 20 or I per sample set	%R = 80-120 RSD < 20%	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Reanalyze.
		Surrogate Spike	All blanks, standards, QC samples, field samples	See method or laboratory controls for surrogate spiking compounds control limits	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
		MS/MSD	1 per every 20 samples	%R = 80-120 RSD = < 20%	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

					•
ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
			:		CORRECTIVE ACTION
EPA 1613	Dioxin	FIELD OC:	·		
		Duplicate	1 for every 10 field samples collected	Water Samples - RPD < 20% Soil Samples - RPD < 35%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed.
		Field Reagent Blank	l per cooler	Less than reported detection limit	Qualify data.
		Rinsate (c)	1 for every 10 field samples collected	Less than reported detection limits	Qualify data.
		LABORATORY QC:			
		Initial Calibration	Prior to analysis and when continuing calibration criteria fail	5 pt. calibration; RSD of mean < 20%	Recalibrate instrument.
		Continuing Calibration	Every 12 hours	Response factors deviate < 20% from average of initial calibration.	Rerun continuing calibration. If still out of control, take remedial action and recalibrate instrument.
		Laboratory Reagent Blank	Each sample set	Less than reported detection limit	Step 1: Reanalyze. Step 2: If second blank exceeds criteria, clean the analytical system. Step 3: Document the corrective action taken and qualify all associated data.
		QC Check Standard (External Source)	Quarterly	%R = 80-120 RSD < 20%	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Reanalyze.
		Sensitivity Check	Every 8 hours of operation	Ion abundance criteria; see method	Tune instrument; repeat.
		Mass Calibration	Every 8 hours of operation	See method	Tune instrument; repeat.
		Laboratory Fortified Blank	Every 8 hours of operation	%R = 80-120 RSD < 20%	Step 1: Reanalyze. Step 2: If still out of control, perform and document remedial action. Step 3: Reanalyze.
		Surrogate Spike	All blanks, standards, QC samples, field samples	Laboratory specified surrogate spiking compounds and control limits	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
		MS/MSD	l per every 20 samples	%R = 80-120 and RSD = < 20% for isotope dilution; or %R = 65-135 and RSD = < 35% for Internal Standard method	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
		•			CONTROL ACTION
EPA 632	Fluometuron	FIELD OC: Duplicate Rinsate (c)	Every 20 samples Every 20 samples	RPD: 35% ≤ detection limit	Flag data.
		LABORATORY OC:		2 detection infilt	Flag data.
		Calibration	At start of analysis	5 pt., average RF can be used if RSD ≤ 10% or use calibration curve.	 Check standard solution. Check system as per manufacturer's instructions.
		Calibration Stability	At end of analysis	CCC response factor ≤ 10% difference from the midpoint standard	 Perform appropriate instrument maintenance. Check standard solution. Check system. Recalibrate and reanalyze samples.
		Method Blank	I per batch of samples analyzed together, minimum I per 20 samples	≤ PQL	 Check system. Check blank. Flag data.
		Laboratory Fortified Blank MS/MSD Duplicate		%R = 77-123	Reanalyze. If still out of control, perform and document remedial action.
			Military and a second	%R = 77-123	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
	Supricate	Minimum 1 per 20 field samples	RPD: ≤ 30%	Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.	

SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES AND QC ACCEPTANCE CRITERIA

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL			
(4,0)	TAGAMETER	CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
					TO THE HOLD
SW846 8330	Explosives	FIELD QC:		•	
		Duplicate	1 for every 10 field samples collected	Water Samples - RPD < 20% Soil Samples - RPD < 50%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether
		Rinsate (c,)	1 for every 10 field samples collected	Less than reported detection limits	proper collection procedures were followed. Qualify data.
		LABORATORY OC:	•	·	
		Initial Calibration	Prior to analysis and when continuing calibration fails criteria	5 pt. calibration, % RSD must be < 20% for all analytes	Recalibrate instrument
		Continuing Calibration	Daily	Response factor must be < 15% from average of initial calibration	Rerun continuing calibration. If still out of control, recalibrate instrument
		Method Blank	Daily	Compounds must be below respective detection limits	Step 1: Reanalyze Step 2: If second blank exceeds criteria, clean the analytical system Step 3: Document the corrective action taken and qualify
		Surrogate Spike	All blanks, standards, QC samples, field samples	See laboratory controls	all associated data. Determine cause (if possible), correct and reanalyze. If cause cannot be determined, flag data.
		MS/MSD	I per every 20 samples	Recovery 25-134%; RPD < 40%	Step 1: Reanalyze extract Step 2: If still out, respike and reanalyze. Step 3: If still out, run LCS. If LCS is good, qualify data. If not, stop analyses until problem corrected.

SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES AND QC ACCEPTANCE CRITERIA

ANALYTICAL METHOD (a,b)	PARAMETER	QUALITY CONTROL CHECK	EDEOUSVOV		
		J. J	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW846 8290	High and d		•		
GC/MS	High resolution PCDDs (dioxins) and PCDFs	FIELD OC:			
		Duplicate	1 for every 10 field samples collected	Above 10X detection limit, % RPD must be less than current control limits: Aqueous samples-RPD < 20%; Non-aquesous samples - RPD < 35%	Review lab QC data to determine if they are in control. In not in control, flag data. Use data to evaluate whether proper collection procedures were followed. If not, determine futher corrective action.
		Rinsate (c)	1 for every 10 field samples Less than reporting limit	Less than reporting limit	Qualify data.
		LABORATORY OC:		•	Quanty data.
		Tune with PFK	At beginning of each analytical run and every 12 hour period during analyses.	Tune to conditions as shown in Table 6 of Method.	Tune instrument; repeat. If cannot correct, reject affected samples.*
		Initial Calibration	5 pt. calibration for each compound in Table 5 of Method.	 %RPD of mean RRF for unlabeled stds. ≤ ± 20% and ≤ ± 30% for unlabeled. S/N for GC signals in SICP ≥ 10. Ion abundance criteria from Table 8 of Method must be met. 	Correct, if possible. If not possible, contact USACE.
		GC Column Performance Check Solution	During initial calibration and at end of every 12-hour shift	 Chrom. separation between 2,3,4,8-TCDD & other unlabeled TCDD isomers resolved with valley of ≤ 25%. 10 seconds or greater tolerence for absolute RT for all components 	Corrective action. Reanalyze extracts of positive samples Reject sample data if not completed.
		Continuing (Routine) Calibration	At end of every 12-hour shift.	%RPD of mean RRF for unlabeled stds. $\le \pm 20\%$ and $\le \pm 30\%$ for unlabeled. Ion abundance criteria in Table 8 of Method must be met.	Correct, if possible. If not possible, contact USACE. If no ave. RFs are needed as per par. 8.3.2.4 of Method, recalibrate, otherwise reject data.
		Static Resolving Power	Beginning and end of each 12-hour shift.	Power at least 10,000 (10% valley definintion)	Implement corrective action. Reanalyze extracts of positive samples. Reject otherwise.
		Method Blank (d)	1 for every 20 samples or extraction batch.	Less than PQL. *	Re-analyze blank. Qualify data appropriately.
		Duplicate	1 per 20 samples or batch.	≤25% RPD.	 Check instrument performance. Check errors in weighing. Review analytical procedures. If problem not located, professional judgement.
		MS/MSD	1 per 20 samples or batch	40-135% recovery.* ≤20% RPD.	If an assignable cause, correct and respike.*
		% Recovery of IS	Each sample.	40-135%	Monitor and correct if further deterioration is noted.*

^{*} Not specifically found in written method.

(a) Reference: Methods for the Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised, March 1983 or Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA 600/4-82-057, July 1982.

(b) Reference: Test Methods for Evaluating Solid Waste, USEPA SW846, 3rd Edition, November 1986, with Updates II and IIA.

(c) Rinsates will not be collected if dedicated sampling equipment is used. Rinsates are not applicable for non-aqueous matrices.

Additional QC may be implemented per manufacturer's instructions.

Appendix B

EMSL ANALYTICAL, INC.

Outline of the LABORATORY QUALITY ASSURANCE PROGRAM

For:

PHASE CONTRAST MICROSCOPY
TRANSMISSION ELECTRON MICROSCOPY
POLARIZED LIGHT MICROSCOPY

The Quality program at EMSL is built on a commitment to quality and continued improvement. This program is a primary part of our every day work: developed, utilized, and maintained by all the dedicated staff at EMSL.

Introduction:

This Program Outline provides a comprehensive overview of the Quality Assurance Program. It provides the reader with a summary of the Laboratory policies and procedures as they relate to the technical aspects of Corporate Quality objectives.

This program follows quality guidelines as documented by the American Industrial Hygiene Association (AIHA), the EPA's National Voluntary Laboratory Approval Program (NVLAP) and other applicable state and federal regulatory agencies.

This QA program is designed to ensure that the highest level of quality professional services and technical excellence is provided to our clients. This is accomplished by the implementation of program policies including:

- Development of company standard quality control programs
- Standardization of reporting formats
- Review of regional laboratory QC performance
- Providing technical training for all staff levels
- Achieving traceability of data
- Performance of quality audits
- Participation in applicable Accreditation Programs
- Participation in applicable third party proficiency testing programs

The objectives of these program polices ensure the quality, accuracy and integrity of our analytical data.

The Quality Assurance objectives, policies and procedures are formally documented in the Quality Assurance Manual – EMSLQAASB100.0. An outline and summary of this manual is presented on the following pages.

General and Administrative

Scope

The Objectives of this Manual are to ensure the following:

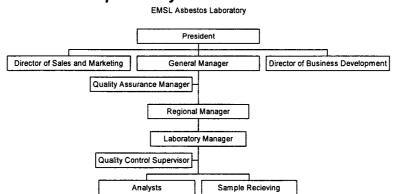
- Quality and accuracy of analytical results.
- Conformance with all analytical methodologies
- Conformance with Corporate mandated QA/QC requirements.
- Delivery of the highest quality of professional services and technical excellence to our clients.
- Ensure data integrity

To achieve these goals, this Manual directs implementation of the Quality Control program and describes responsibilities and duties of all personnel, and addresses all aspects of Quality Assurance for phase contrast microscopy (PCM), transmission electron microscopy (TEM) and polarized light microscopy (PLM) laboratory operations.

This Manual is to be kept accessible to all employees, and all employees are responsible for being familiar with, and adhering to its contents. Each employee is to sign the signature page acknowledging an understanding of the contents of this document. A copy of this signature page is submitted to the QA Department.

This Quality Assurance Program will be reviewed at least annually by the QA Manager. It will also be reviewed any time a problem arises that indicates a possible program flaw. In such an instance, the QA Manager will discuss the problem with Regional and Laboratory Management, Quality Control Supervisor and Analysts to ensure needed input from all levels within the Laboratory.

Organization and Responsibility



Ethics

One of the objectives of the Quality Assurance Program is to insure the staff of EMSL is provided training in the aspects of ethics as they pertain to corporate policy. The goals of this training program are:

- For each staff member to understand the responsibility to provide true and accurate information
- The understanding of the consequences of questionable conduct
- Provide direction to employees regarding ethics issues
- Provide support to employees regarding ethics issues
- Define right and wrong (as it is job related)
- The understanding of the impact of our actions

Training will be provided in the form of workshops, required readings and Corporate issued news letters. The Quality Assurance Department is responsible for insuring that this training is provided to the staff and that records are maintained documenting such training.

Standard Operating Procedures

Technically specific Operating Procedures are documented in the SOP manuals, located at each laboratory facility. These SOPs include step by step procedures for the preparation, analysis, and reporting of data. These documents are controlled by the QA Department and include:

EMSL.XXTEMSOP.200.x - Standard Operating Procedures for Transmission Electron Microscopy EMSL.XXPLMSOP.200.x - Standard Operating Procedures for Polarized Light Microscopy EMSL.XXPCMSOP.200.x - Standard Operating Procedures for Phase Contrast Microscopy EMSL.QCPRGMSOP.200.x - Standard Operating Procedures for the Quality Control Program EMSL.QAAUDITSOP.200.x - Standard Operating Procedures / Quality Assurance Audits

These SOPs cover methodology for analytical procedures, calibrations, contamination checks Quality Control frequency, procedures, and internal audit policies

Each analytical SOP (TEM, PLM, and PCM) is edited specifically for the laboratory operation. The Laboratory Manager is responsible for insuring the SOP's reflect the actual laboratory procedures and are reviewed and updated annually.

Sample Tracking

Rigorous sample tracking is fundamental to a QA Program. The most thorough and complete analysis is useless if performed on the wrong sample.

Our sample-tracking program is designed, to the extent that it is possible, to meet all litigation requirements. It is also designed to have redundancy safeguards wherever possible.

In order to ensure the integrity of any sample, records of its custody must be maintained throughout the sample collection in the field, acceptance by the laboratory, and analysis.

A sample will not meet litigation requirements without a chain of custody that begins at the sample collection point. Since the client collects samples for analysis, the laboratory cannot be responsible for issuing a chain of custody at the time of sampling. However, the laboratory will advise all clients regarding sampling requirements (sampling materials, recommended sampling volumes, packaging, instructions for shipping, etc.)

and chain-of-custody, and recommend that they use our form if they do not have their own.

The chain-of custody form will include:

- Analysis Required
- Date of Sampling
- Location of Sampling (if supplied)
- Sample volume (if supplied)
- Unique sample ID for each sample submitted
- Date submitted to laboratory
- Record of Custody

Prior to accepting samples, the Sample Receiving Coordinator inspects them to determine if they conform to laboratory acceptance criteria. If they do not, or if the clerk has any question as to the validity of the sample, the Laboratory Manager or an analyst trained to analyze such samples will determine whether the damage to integrity is sufficient to cause rejection. Rejections of samples are to be followed up by immediate notification of the client with an explanation and return of the questionable sample, if required by the client.

Samples are judged unacceptable under the following circumstances:

- Analysis requested outside laboratory's scope of accreditation
- Analysis requested outside laboratory capability (such as lack of equipment or staffing resources).
- Obviously damaged or compromised samples, i.e. opened air cassettes, cassettes with torn or ripped filters, water samples in leaking or faulty containers.
- Improper labeling
- Improper packaging
- Impossible deadlines
- Obvious faulty sampling technique
- Improper sample media
- Incompatible samples packaged together (i.e.- air samples with bulk samples)
- Inappropriate analytical methodology requested

Log in of samples is normally done by the Sample Receiving Coordinator, but may be done by any other employee familiar with the process. Information is entered for samples received into the Laboratory Information Management system (LIMS). LIMS is a computer laboratory management system which serves to track all samples from receipt through the analysis, reporting, and billing processes. Access to the LIMS system is restricted to approved personnel only. The Laboratory Manager is responsible for assigning computer rights to all applicable personnel and is accountable for ensuring that sound security measures are maintained.

The Sample Receiving Coordinator inspects the samples for integrity, verifies that all samples listed on the chain of custody are present, and logs them into the computer system. Any damages are noticed, and are reported to the laboratory manager.

All analyses must be carried out in accordance with the SOP(s) indicated. All SOPs used in this Laboratory will be found in the EMSL Laboratory Standard Operating Procedure Manuals.

Data Recording

Once analysis of a sample has been completed, the analyst signs the analytical worksheet and any other appropriate documentation. Chain of custodies and analytical worksheets are copied and placed in the laboratory master files. Originals are submitted to the Laboratory Manager for review and approval, before preparation of a client-ready report. All records are to be retained for a minimum of 7 years or as requested by the client.

Analytical data storage, processing, and reporting is facilitated through use of Laboratory Information Management System (LIMS) computer software. When samples are received by the laboratory personnel, sample information is entered into the LIMS system, which assigns the batch of samples a unique project identification number and generates analytical worksheets. The samples and worksheets are then forwarded to the analysts to be analyzed.

Once sample analysis has been completed, this result data is entered into the LIMS software. Analytical result data is entered either by approved data entry personnel, or by the analysts themselves. The LIMS software stores the analytical data, performs calculations where applicable, and generates the final report for the project. This final report is reviewed and approved before being forwarded to the appropriate client.

Archival and Disposal of Samples

Once the analysis is complete and the analysis worksheet is signed, the analyst stores the sample in the appropriate storage box, as indicated in the SOP. All storage boxes are to be stored in a safe manner for the period indicated for that category of waste, in accordance with regulatory requirements. When a storage box is full, the month of which the samples where analyzed (or similar reference numbering system as appropriate for the operations, i.e. billing number) is marked on it. A new storage box replaces the old one which is then to be stored until time of disposal.

All bulk and air samples are held for a minimum of 3 months, unless a longer period is requested by the Client. All TEM grids are held for 3 years. Asbestos containing samples are disposed of by a licensed contractor, and a copy of the waste manifest is obtained and kept on file. If requested, samples will be returned to the Client.

Quality of Materials

The high quality of materials used in this Laboratory shall be assured through specific purchasing and verification procedures and/or proper preparation techniques.

Selection of the appropriate grade of reagent(s) is designated in the reagent section of each analysis SOP and in addition may be specified by the Laboratory Manager in unusual circumstances. As a general practice, reagents will be of at least ACS reagent quality.

Reagents inclusive of SRM shall be purchased in accordance with the analytical needs of this Laboratory as determined by the Laboratory Manager. When received by the laboratory, these item's labels are dated and initialed with date received and expiration dates (if appropriate) as indicated /suggested by the manufacturer. Labels are also dated and initialed when opened and/or when reagent mixtures are prepared.

Verification will consist of confirming that the priority grade recorded on the reagent label conforms to the requirements of the SOP unless analysis difficulties indicate a possible problem or regulatory agency requirements specify otherwise. In the latter case, the appropriate analytical SOP will indicate the proper verification procedure.

Equipment/instrument maintenance

Maintenance schedules for equipment will be established by the Laboratory Manager. The Laboratory Manager shall also determine whether each microscope is maintained and repaired in-house or by an outside agency following EMSL administrative procedures. Servicing will also be performed when a need had been identified by calibration or other QC checks.

A maintenance file will be maintained for all equipment. In addition to a schedule of normal preventive maintenance, this file will contain a record of servicing.

Contamination Management

This Section describes reagent control, contamination management, and use of controlled procedures for this Laboratory. Proper observance of laboratory procedures is necessary to guarantee accuracy of results and the safety of Laboratory staff members.

Contamination both of samples and of the environment (including reagents used in analysis) must be avoided to provide the highest quality, legally defensible data to our clients. In order to achieve this goal, Laboratory staff must adhere to various preventative measures and use the testing procedures for contamination detection as established by the QA Manager.

If analysis of the blank samples indicate the possibility of contamination, the area and tools are cleaned and another slide prepared and analyzed. If the second slide shows contamination, applicable reagents are checked (acetone, triacetin, dispersion oils, etc.). A new box of slides is used to prepare a third slide. If analysis of the third slide shows contamination, a complete investigation is conducted to determine the contamination source.

If contamination is detected in any situation, the source of contamination must be traced and the problem resolved to prevent reoccurrence. All procedures taken to resolve a contamination circumstance shall be documented properly and completely in the laboratory files.

Document preparation and control

In order to prepare and distribute documents in an organized fashion, procedures for initiation, preparation, review, approval and issuance of controlled copies will be followed. This program is a coordinated effort involving both technical review and custodial control. Analysts are to use only controlled, i.e., approved documents for all calibrations, analyses, final reports, and other activities performed in this laboratory.

Reporting results

All final client reports are to be reviewed, approved, and signed by the Laboratory Manager prior to being sent to the client. They are also subject to review by the QA Manager.

Results are cleared for reporting by quality control data review and confirming analysis.

Quality Control statistics shall be reviewed on a regular basis as determined by the QA Manager in accordance with regulatory agency requirements. Specific Quality Control procedures are detailed in the 'Performance Criteria' sections for each of the Method Modules found in this document. In general, 10% of analyses are reanalyzed using various QC procedures as appropriate for the methodologies. Samples are chosen randomly. The analyst records their reanalysis results on the data sheet in addition to notes whether any serious discrepancy exists. The Laboratory Manager periodically reviews the data sheets and the reanalysis data. If the difference between analyses is within control limits for QC analysis, the results will be cleared for reporting. As long as those statistics are deemed acceptable, client reports will continue to be processed.

If the difference between analyses exceeds control limits the Laboratory Manager and the analyst will review the sample data and resolve the differences. A detailed corrective action report recording all activity is submitted to the QA Manager. (See Procedures for Dealing with Deficiencies, Section 9.0)

In addition to QC review, analytical data is reported with confidence based on compliance with this QA program. The traceability of the data reported is insured through the procedures and policies as documented in this manual, including:

- Delineation of Responsibility
- Compliance with Analytical Standard Operating Procedures
- Following Calibration Protocols
- Fulfillment of the Required Amount of Quality Control Analysis
- Satisfaction of Training Requirements

Records Retention

The following records shall be maintained for 7 years:

- Copy of Chain of Custody Documents
- Original Analytical Data Recording Worksheets
- All other records relating to the preparation of the client report

Procedures for dealing with deficiencies

Any complaint by a client will be treated as a non-conformance, and treated with the same corrective action follow-up as a discrepancy seen in following internal Quality Control procedures.

If a client makes a complaint about a test result, the sample in question will be reanalyzed by a second Analyst. If the second result agrees with the original the Laboratory Manager shall advise the client in writing that a quality control check has confirmed the original analysis.

In all cases where a deficiency is discovered, the QA Manager will initiate a corrective action review to determine the root cause of the problem and action to take to prevent reoccurrence. A report will be issued to the Laboratory Manager, who is responsible for the corrective action implementation.

The corrective action will consist of a review of all steps leading up to the non-conformance. This will include review of QC data, sample tracking, data transcription, instrument calibration, training documentation, and discussion with personnel.

Following the review, the QA Manager will prepare a report detailing the cause of the error and corrective action to take to prevent re-occurrence. The QA Manager will also

follow up on the corrective action to ensure its implementation

Analytical Performance Criteria

Performance criteria will be determined three ways:

- 1) Results from intra-lab and round robin testing will be plotted to see if they fall within warning and action limits.
- 2) The administering agencies for proficiency testing will determine performance criteria.
- Achievement of internal on-site Quality audits by the Regional or QA Manager. These audits will verify compliance with all QA and QC policies as documented in this manual. The Quality Audit process is detailed below.

Quality Control is performed continuously throughout the course of laboratory sample analysis regardless of laboratory productivity and is made part of the normal course of laboratory sample analysis. Frequency and volume of QC analysis is based on regulatory requirements and Good Laboratory Practice. These requirements are listed for each analysis type in Appendix A of this manual.

These methods will be used according to the scope of the laboratories accreditation status and quality control requirements for each type of analysis. Performance criteria will be maintained for both individual analysts and for the entire laboratory. The standards for acceptance criteria are documented in the EMSL Quality Control Standard Operating Procedure Manual, *EMSLQCPGRMSOP.200.x.*

Quality Audits

Quality Audits will be performed for each laboratory location on an annual basis or more frequently as deemed necessary by Corporate, Regional or Laboratory Management. Audit procedures and policies are issued by the Quality Assurance Department and include:

- Review of compliance with the Quality system
- Compliance with Quality Control analysis
- Identification of any problem areas and suggestions for resolution

The Quality Assurance Department develops the guidelines and overall manner by which a Quality Audit is performed. These policies are detailed in the Standard Operating Procedure for Quality Audits, *QAASBAUDITSOP.200.0*.

Proficiency Testing Programs

Laboratories participating in proficiency testing programs will insure the analysis is performed using the same analytical methodology and staff as under normal, client sample conditions. At no time is there inter-laboratory exchange of samples.

Records of proficiency testing analysis are to be completed and maintained in a separate laboratory PT file. This data is also maintained for each participating analyst in his or her personal training file.

Analytical Quality Control Programs

The Quality Control program as established and managed by the QA Manager ensures that this Laboratory produces quality data. This process ensures, at a minimum, that our data is legally defensible and that all personnel perform their responsibilities properly. The Laboratory Manager will determine how QC testing is implemented operationally (e.g., after the analyses of every ten samples or at the end of each day, etc.) QC analysis is performed on a minimum of 10 % of sample volume. QC testing occurs on a regular basis and is not scheduled around the amount of workload.

In addition, the QA Manager will inspect the results of all QC testing on a regular basis and provide the necessary support and directives to the Laboratory Manager to ensure the QC program is properly executed.

Quality Control - General

Our laboratories internal QC program includes at a minimum, 10% quality control on all samples received for analysis. These are summarized below in each analytical section and include:

- Analysis of standard reference materials
- Intra analyst QC
- Inter analyst QC
- Analysis of blank samples
- Participation in inter laboratory programs
- Participation in proficiency testing programs

This QC data is graphed on control charts designed specifically for each analysis type. The description of these control charts are detailed in the Standard Operating Procedure EMSLQCPROGSOP.200.

The Laboratory Manager (or Managers designee, i.e. QC Supervisor) of each laboratory is responsible for implementing the day-to-day QC testing and ensuring the correct types of testing occurs at the appropriate frequencies. The Laboratory Manager is also responsible for ensuring complete records of QC testing are maintained.

Training - General

New analysts with no prior formal training must complete the EMSL training program in asbestos analysis in order to perform such analysis independently. The Lab Manager will draw on the candidate's previous training, if any. The candidate will receive sufficient in-house training to demonstrate proficiency and understanding in all related topics to the Lab Manager's satisfaction.

Practical Factors: When the candidate has received sufficient training to analyze samples, he/she will work in the laboratory along side an experienced analyst. The candidate will not sign any reports. All samples will be checked by an experienced senior analyst, who will officially report the results for review and signature, by the Laboratory Manager.

Proficiency Analysis: The candidate will be deemed proficient when quantitation within I laboratory norms, as established by the QC schedule are met.

Additionally, the trainee must perform analysis on past proficiency samples and succeed in generating data within the acceptable range as established by the agency(ies) statistical analysis. (see training SOP for additional detail)

Records are kept of the candidate's progress by the analyst-training log. When all areas are signed off, the candidate may perform independent analysis.

Phase Contrast Microscopy (PCM) Method following: NIOSH 7400

The Quality Control program for phase contrast microscopy includes intra-analyst sample testing, participation in inter-lab programs, and statistical evaluations and calibrations.

Calibration

Calibration procedures must be followed prior to the analysis of air samples to insure that results of analysis reflect true and accurate data. The following summarizes the type and frequency of calibration for the analysis of fibers in air by PCM. Details of these procedures are found in the PCM SOP, *EMSL.XXPCMSOP.200.x.*

1) Microscope calibration

Phase Ring Alignment Contamination control HSE/NPL Test Slide Measurement of Walton Beckette Graticule

- 2) Analysts calibration
 Standard reference slide (past Proficiency test slide)
- Operational Calibration
 Air monitoring
 Hood calibration

Details on the calibration procedures for PCM can be found in the Standard Operating Procedures Manual.

Quality Control Analysis

Each analysis performed in this Laboratory has its own Quality Control requirements that have been determined by the QA Manager and are delineated in that SOP. These requirements include but are not limited to the following:

Intra-Analyst
Reference
Proficiency Testing
Round Robin Testing
Laboratory Blank Analysis

The Laboratory Manager (or Managers designee, i.e. QC Supervisor) of each laboratory is responsible for implementing the day-to-day QC testing and ensuring the correct types of testing occurs at the appropriate frequencies. The Laboratory Manager is also responsible for ensuring complete records of QC testing are maintained.

Transmission Electron Microscopy (TEM)

Method following: AHERA - 40 CFR, Part 763, Subpart E, EPA Level I, II, III - EPA Contract # 68-02-3266, ASTM D 5755-95

The QA/QC program for the analysis of asbestos via TEM insures compliance with standard regulatory guidelines and follows Good Laboratory Practice (GLP). The program includes:

- Achievement of Verified Status
- Classification of Structures
- Calibrated Measurements at .5 micron
- Calibrations
 - -alignments
 - -magnification
 - -camera constant
 - -plasma asher
 - -detector resolution
 - -grid opening measurements
 - -analytical balance
 - -muffle furnace
- Fiber Id and Sizing
- SAED Indexing
- Ambient air monitoring

Quality Control Analysis

Each analysis performed in this Laboratory has its own Quality Control requirements that have been determined by the QA Manager and are delineated in that SOP. These requirements include but are not limited to the following:

Intra-Analyst
Intra-Analyst repreparation
Inter-Analyst
Reference Standards
Verified Analysis
Proficiency Testing
Round Robin Testing
Laboratory Blank Analysis

Polarized Light Microscopy

Method following: EPA-600/R-93/116, EPA-600/M4-82-020

Quality control procedures in the PLM laboratory follow guidelines as documented by the NVLAP accreditation program.

Calibration procedures must be followed prior to the analysis of samples to insure that results of analysis reflect true and accurate data. The following summarizes the type and frequency of calibration for the analysis of asbestos in bulk materials by PLM. Details on the performance of these functions are found in the PLM SOP.

1) Microscope calibration

Center Stage or objective, & condenser Align polars Crosshair alignment fixed in polarizer's privileged direction

2) Analysts calibration

Standard reference sample
Contamination check with fiberglass sample
Check Standard Amosite mount for proper dispersion colors,
refractive index

3) Operational

Calibrate Analytical Balance
Air monitoring
Refractive mounting oil calibration
Calibrate muffle furnace temperature
Hood calibration

Details on the calibration procedures for PLM can be found in the Standard Operating Procedures Manual.

Quality Control Analysis

Each analysis performed in this Laboratory has its own Quality Control requirements that have been determined by the QA Manager and are delineated in that SOP. These requirements include but are not limited to the following:

Intra-Lab Testing
Inter-Analyst
Intra-Analyst
Reference Standards
Proficiency Testing
Round Robin Testing
Laboratory Blank Analysis

United States Department of Commerce National Institute of Standards and Technology



ISO/IEC GUIDE 25:1990 ISO 9002:1987

Certificate of Accreditation



EMSL ANALYTICAL, INC.

WESTMONT, NJ

is recognized under the National Voluntary Laboratory Accreditation Program for satisfactory compliance with criteria established in Title 15, Part 285 Code of Federal Regulations. These criteria encompass the requirements of ISO/IEC Guide 25 and the relevant requirements of ISO 9002 (ANSI/ASQC Q92-1987) as suppliers of calibration or test results. Accreditation is awarded for specific services, listed on the Scope of Accreditation for:

BULK ASBESTOS FIBER ANALYSIS

<u>June 30, 2001</u>

Effective through

Pavid F. alderman

For the National Institute of Standards and Technology

NVLAP Lab Code: 101048-0



NEW YORK STATE DEPARTMENT OF HEALTH

ANTONIA C. NOVELLO, M.D., M.P.H. Commissioner



Expires 12:01 AM April 1, 2001 ISSUED April 1, 2000 REVISED June 29, 2000

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

Lab ID No.: 10872

Director: DR. PETER FRASCA
Lab Name: EMSL ANALYTICAL
Address: 107 HADDON AVE
WESTMONT NJ 08108

is hereby APPROVED as an Environmental Laboratory for the category

ENVIRONMENTAL ANALYSES/ POTABLE WATER

All approved subcategories and/or analytes are listed below:

D.W. Miscellaneous : Asbestos

Serial No.: 106751

Wadsworth Center

Property of the New York State Department of Health. Valid only at the address shown. Must be conspicuously posted. Valid certificate has a red serial number.

NEW YORK STATE DEPARTMENT OF HEALTH

ANTONIA C. NOVELLO, M.D., M.P.H. Commissioner



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WESTMONT NJ 08108

is hereby APPROVED as an Environmental Laboratory for the category

ENVIRONMENTAL ANALYSES/SOLID AND HAZARDOUS WASTE

All approved subcategories and/or analytes are listed below:

scellaneous : Asbestos in Friable Material Asbestos in Non-Friable Materia

Serial No.: 106753

Wadsworth Center

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DOH-3317 (3/97)

II

DETERMINATION OF ASBESTOS IN BULK SAMPLES BY POLARIZED LIGHT MICROSCOPY (PLM) WITH DISPERSION STAINING

1.0 OVERVIEW

- 1. This method describes the procedures for the determination of the presence or absence of asbestos in bulk samples of building material. Samples are initially examined under low magnification using a stereo microscope, contained in a hood equipped with a HEPA filter. Initial observations should note gross material appearance (homogeneity, fibrous/non-fibrous) and physical characteristics (color, texture, friable/non-friable).
- 2. Analysis by polarized light microscopy (PLM) is used for the positive identification of suspect fibers. Positive identification of asbestos requires the determination of several optical properties peculiar to the six types of asbestos: chrysotile asbestos, grunerite asbestos (amosite), riebeckite asbestos (crocidolite), anthophyllite asbestos, tremolite asbestos and actinolite asbestos.
- Quantitative estimates of the asbestos content, and other major constituents, of the sample are made based on a combination of the estimates from both the gross and the PLM examinations.
- 4. Interference's from other inorganic and organic fibrous constituents, cleavage fragments of natural minerals, binders, coatings, and man-made fibers may be encountered. Moisture may interfere with the determination of some optical properties. Therefore, wet samples should be dried prior to analysis.
- 5. The sample matrix may cause a variety of interference's under PLM observation. Special matrix reduction techniques may be necessary to reduce these interference's.

2.0 EQUIPMENT

- 1. A low power binocular microscope (preferable stereomicroscope), with a magnification range of approximately 10-45X, and an auxiliary light source.
- 2. A compound microscope set-up for polarized light microscopy, to include a polarizer, analyzer, port for a wave retardation plate, a 360° graduated rotating stage, substage condenser, lamp and lamp iris.
 - Objective Lenses: 10X, 20-25X, 40-45X, and dispersion staining objective.
 - Ocular Lens: 10X minimumEyepiece reticule: Cross hair
 - Compensator plate: 550 millimicron retardation (first-order red or gypsum)
- 3. The type of material being examined will dictate the various apparatus needed for sample preparation. At a minimum, the following will be required:
 - Negative pressure hood equipped with a HEPA filter at the exhaust
 - Microscope slides: ~75 mm x 25 mm, 1 mm thickness
 - Coverslip: No. 1, 22 mm²
 - Tweezers, tungsten probes, dissecting needles, scalpels, glazing pliers, forceps.
 - Glass plates, petri dishes or disposable containers(e.g. weighing boats 5"²)
 - Mortar and pestle (agate or porcelain)
- 4. Auxiliary equipment may include a Wylie mill, centrifuge, filtration apparatus, and low temperature ashers, assorted beakers, and miscellaneous glassware, a vacuum cleaner equipped with a HEPA filter.

3.0 REAGENTS

- 1. Refractive index liquids
 - N_D = 1.550, 1.605, 1.630, 1.680, 1.700

2.

- Dilute acetic acid (CH₃COOH): ACS reagent grade
- Dilute hydrochloric acid (HCI): ACS reagent grade
- Acetone (CH₃COOH₃): ACS Reagent grade
- Chloroform (CHCl₃): ACS Reagent grade
- Asbestos reference standards, and standards for various minerals and man-made materials typically encountered in bulk materials containing asbestos. Use NIST Certified SRM 1866a/Common Commercial Asbestos, SR1867/Uncommon Commercial Asbestos.

4.0 BACKGROUND AND DEFINITIONS

The name asbestos, a Greek word mistakenly thought to mean incombustible, was given to fibrous minerals hundreds of years before the science of mineralogy evolved. The Greek word actually means unquenchable, inextinguishable (not incombustible) according to the etymology of the Oxford English Dictionary.

The definition of asbestiform minerals includes three aspects: morphology, structure, and chemistry. Morphologically, asbestiform mineral varieties separate into flexible fibers or flexible bundles of fibers. Flexible fibers bend readily and only break across the fibers into distinct pieces with some difficulty. Structurally, the asbestiform minerals are limited to the serpentine and amphibole mineral groups. Chemically, these minerals are all hydroxylated silicates. The term "hydroxylated" is preferred over "hydrated" because these minerals contain OH ions rather than water or crystallization. The serpentines contain approximately 13-weight percent water; and the amphiboles, approximately 2.5 weight percent water.

There is no "group" of asbestos minerals. "Asbestos" is a general term applied to certain minerals (which are themselves classified under crystal-structure-based groups) when these minerals crystallize as the asbestiform variety. Table 2 lists some common silicate minerals and their asbestiform varieties, together with their relationships and formulas in Tables 3 & 4.

Only very small quantities of the amphibole and serpentine minerals under particular geological circumstances occur as an asbestiform variety of the mineral. The asbestiform varieties occur in veins or small veinlets within rock containing or composed of the common (nonasbestiform) variety of the same mineral.

In some rare instances, the mineralogical occurrences contain sufficient quantities of usable asbestiform minerals to be economically mined for commercial asbestos. The soft, silky fibers of asbestos (sometimes called mineral silk) are so flexible that they can be spun into threads from which cloth can be woven. The resulting material is fireproof, is a good thermal and electrical insulator, and has moderate to good resistance to acids. It has been used from Roman times, and is most familiar in daily use in brake lining for automobiles and as the "asbestos" siding used in residential construction.

The six asbestos minerals are defined under two mineral groups:

- 1. The serpentine group and
- 2. The amphibole group.

Serpentine Asbestos

Chrysotile is the only commercial asbestos mineral belonging to the serpentine group. Moderate amounts of aluminum may substitute for silicon and moderate amounts of iron may substitute for magnesium. Small amounts of manganous oxide (Mn)), calcium oxide (CaO), potassium monoxide (K_2O) and sodium monoxide (Na_2O) are also reported in the chemical analyses.

The crystal structure of chrysotile asbestos consists of double layers. Each layer consists of a linked SiO_4 tetrahedral coordinated to a second layer of linked MgO_2 (OH)₄ octahedral through a sharing of oxygen atoms; the composite double layer rolls up (like a window shade) to form long hollow tubes. The diameters of the individual tubes are on the order of 35 mm, and the length-to-diameter ratio can vary from 10:1 to well over 10,000:1.

Chrysotile is characterized by a combination of (1) a distinctive shape, (2) a chemical composition close to $Mg_3Si_2O_5$ (OH)₄, and (3) characteristic X-ray and electron diffraction pattern.

Amphibole Asbestos

Five of the six commercial asbestos minerals belong to the amphibole mineral group. These are grunerite asbestos (usually but improperly referred to by the acronym amosite); riebeckite asbestos (usually referred to by the variety name crocidolite); anthophyllite asbestos, tremolite asbestos; and actinolite asbestos. A considerable amount of substitution of other elements for Fe²⁺, Fe³⁺, silicon, sodium, calcium, and magnesium can take place in these minerals.

The Crystal structures of the amphibole minerals, including the asbestiform varieties, are composed of strips or ribbons of linked polyhedra, which join to form the three-dimensional crystal. The individual stripes are composed of three elements: These are two double chains of linked (Si, Al)O₄ tetrahedral and a strip of linked MgO₆, FeO₆ or AlO₆ octahedral.

4.1 Properties of Asbestos

Asbestos is a fibrous mineral of unique properties. It is used in a multitude of different applications because it can confer superior properties on products, including the following:

- stability in resistance to heat, moisture and microorganisms:
- insulation against noise, heat and electricity
- resistance to wear and to deformation under load or impact
- improved smoothness, hardness and opacity
- · resistance to chemical attack, leaching and decay.

4.2 Asbestos Related Terms

In the following discussion, asbestiform refers only to asbestos. The other term, "fibrous", "mineral fiber", "fibril" and "fibril structure" applies to both asbestiform and non-asbestiform varieties.

<u>Asbestos:</u> A collective mineralogical term encompassing the asbestiform varieties of various minerals; an industrial product obtained by mining and processing primarily asbestiform minerals.

The quality of asbestos depends on the mineralogy of the asbestiform variety, the degree of asbestiform development of the fibers, the ratio of asbestiform fibers to acicular crystals of other impurities, and the length and flexibility of the fibers. The major asbestiform varieties of minerals used for asbestos are chrysotile, tremolite-actinolite asbestos, cummingtonite-grunerite asbestos, anthophyllite asbestos, and crocidolite. Asbestos may be marketed by its mineral name such as Amosite or Montasite. Some asbestos products contain non-asbestiform minerals (for example, asbestos-cement and asbestos-magnesia); consequently, the mineralogical and the industrial definitions of asbestos do not always coincide.

<u>Fibrous:</u> The occurrence of a mineral in bundles of fibers, resembling organic fibers in texture, from which the fibers can usually be separated (for example, satin-spar, and chrysotile).

The term "fibrous" has been used during the last 200 years to describe all kinds of minerals that crystallized in habits resembling organic fibers, including asbestos minerals. However, the related term "asbestiform" was never used for fibrous mineral habits other than asbestos. Accordingly, "fibrous" is the more general term, and asbestiform is a specific type of fiber.

<u>Mineral Fiber:</u> The smallest elongated crystalline unit which can be separated from a bundle or appears to have grown individually in that shape, and which exhibits a resemblance to organic fibers. (Examples: fiber bundles, chrysotile and crocodile, individual fibers, epsomite and Millerite).

The term "fiber is not limited to asbestos. However, it is distinct from "acicular" because it requires the resemblance to organic fibers.

<u>Fibril:</u> A single fiber, which cannot be separated into smaller components without losing its fibrous properties or appearances.

Most fibers are single structural entities, such as Millerite and nickel sulfide, and some may be called fibrils. However, some fibers are composed of two or more fibrils that are less readily separable from each other than fibers are from bundles (for example, chrysotile and crocidolite).

<u>Fibril Structure:</u> A systematically deformed and/or defective crystal structure of a fibril. A defect structure would involve various type of dislocation. The fibril structure may be exhibited by a single crystal, a group of single crystals, or at twinned single crystal.

The scroll-like fibril structure of chrysotile, the twinned single crystal fibrils of chrysotile, and the incompletely resolved fibril structure of an amphibole are all examples illustrated in the literature.

Some acicular single crystals may have the appearance of fibers and fibrils, yet there is nothing unusual about their crystal structures. Other acicular single crystals may have significant structural deviation sin addition to appearance which result in the display of certain properties usually found in fibers such as high tensile strength along the fiber axis. Thus, fibril structure is not limited to asbestiform structures, but may occur in a minor form in non-asbestiform structures.

<u>Asbestiform:</u> A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

"Asbestiform" and "asbestos" are essentially synonymous in current usage. Some special properties of asbestiform varieties, including optical extinction and surface charge, are either not fully understood or are not uniformly applicable to all asbestiform fibers; consequently, they cannot be considered fundamental characteristics at this time.

4.3 Commercial Asbestos Minerals

<u>Chrysotile:</u> found in white, wavy, silky, lustrous fiber bundles. The fibers are usually much longer than they are wide. Chrysotile is often found in woven materials because of its flexibility.

Amosite: found in tan-brown, straight, brittle, rigid, inflexible fiber bundles.

<u>Crocidolite:</u> found in blue-blue-gray, straight, rigid fiber bundles. It is probably the most toxic form of asbestos we know.

<u>Anthophyllite:</u> usually colorless to pale brown. It may be found as singly crystals or fiber bundles. Fibrous anthophyllite is generally long and thin.

<u>Actinolite and tremolite:</u> difficult to identify, appear as acicular (bladed) and prismatic (more massive) cleavage fragments.

4.4 Polarized Light Microscopy Terminology

<u>Crossed polarized</u> (polarizer and Analyzer crossed): A fiber is isotropic (has only one refractive index) if it appears black (dark on a dark background) as the stage is rotated. It is extinct at all angles. Such a fiber cannot be an asbestos fiber.

A fiber is anisotropic (has more than one refractive index) if it shows up, as the stage is rotated, alternately light on a dark background

Sign of Elongation: A first order red plate is a section of quartz. It produces a 530nm retardation between the fast ray (X' along the long edge of the plate) and the slow ray (z' along the short edge of the plate). At crossed polars, if the fiber turns yellow in a NW-SE direction (parallel to the red plate port), it displays a positive sign of elongation. If the fiber turns blue when oriented in a NW-SE direction, it displays a negative sign of elongation. Crocidolite is the only asbestos mineral with a negative sign of elongation.

<u>Dispersion Staining:</u> λo is the wavelength at which solid and liquid match in refractive index. Dispersion staining requires "stops" in a special objective. The annular stop allows colors through. The central stop allows complementary (white light $-\lambda o$) colors to pass through. Reference tables exist which show the complementary annular and central stop colors for different asbestos minerals in different immersion liquids. If fiber and liquid Rl's are too far apart, then no dispersion staining colors will result.

<u>Pleochroism:</u> Pleochroism is one of the least reliable asbestos identification characteristics. Pleochroism refers to the tendency of a fiber to change color tint when rotated on the stage in plane polarized light. Most asbestos minerals are nonpleochroic. That is, they do not appear to change color tint as the stage is rotated in plane polarized light. Filler-binder materials contained in the insulation sample, however, may coat the asbestos fiber bundles and create a false pleochroic response. The most strongly pleochroic asbestos mineral is crocidolite, which usual appears to change from a blue to a blue-gray as the stage is rotated.

5.0 ANALYTICAL METHODOLOGY

Note: Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are usually friable and may release fibers during handling or matrix reduction steps. All sample and slide preparations should be carried out in a ventilated hood or glove box with continuos airflow (negative pressure) and a HEPA filtered exhaust. Handling of samples without these precautions may result in exposure to the analyst and contamination of samples and the work environment, by airborne fibers. The cleanliness of the air in the work area is also ensured by testing the air quarterly with TEM analysis.

5.1 Sample Preparation

Gross examination of bulk samples is performed under low magnification (10-45X) to identify homogeneity, layering color, texture, friability and the presence or absence of fibrous constituents.

The sample is carefully removed from the sampling container and placed in an examination disk. Sample integrity is maintained at this point in order to note any layering, and if possible, orientation of the top and bottom surfaces. When discrete layers are identified, each is treated as a separate material, identifying and quantifying fibers in each layer. Each layer is analyzed and reported separately.

All fibrous materials are isolated (subsamples) and prepared for examination by polarized light microscopy. Isolation of these materials results in the loss of sample integrity since the sample must be "picked" through using forceps, probes, and needles. If the sample is not readily friable, a mortar and pestle can be used to crush the sample, or smooth jawed glazing pliers used to break the sample.

The type of sample matrix must be considered when determining sample preparation methodology. In samples such as floor tiles, roofing felts, tars, mastics and chalking, the fibrous materials of interest are often bound in a non-friable, organic substance, which makes observation of asbestos fibers difficult. Special techniques are used to reduce or remove these interference's such as ashing and solvent dissolution. These techniques are detailed below.

5.2 Sub-Sample Preparation

Representative sub-samples of suspect fibrous material must be obtained from a variety of matrix materials. In most cases, forceps and probes are sufficient to isolate fibrous materials for analysis by PLM.

Sub-samples are immersed in an appropriate refractive index liquid on a microscope slide, teased apart, covered with a cover glass, and observed with the polarized light microscope. A refractive index liquid is chosen based on the fiber's morphology as observed under the stereomicroscope.

The selection of appropriate procedures for identifying and collecting subsamples is dependent on the sample matrix. The following are presented as sample preparation steps for typical bulk sample materials.

5.2.9 Soils

"____"

Soil samples present a variety of analytical challenges in that they are not homogenous bulk materials. The standard PLM Methodology (EPA 600/R-93/116) does not provide clear guidelines for the treatment during sample preparation or for the interpretation of the final results. However, a number techniques may be employed by the laboratory. These include:

Standard PLM-The sample is prepared as though it where a bulk building material. Relative homogeneity is assumed. After scanning under the stereoscope, a number of subsample preparations are prepared and scanned under Polarized Light. Quantitation of asbestos fibers is not generally performed due to the lack of uniformity the sample. Sample results are reported as 'none detected' or 'asbestos present.'

MSD 9000- This technique provides for the quantitation of asbestos concentration. This method uses a screening process which facilitates the process of quantitation. See SOP MSD 9000

5.3 Asbestos Identification

Positive identification of asbestos requires the determination of the following optical properties:

- a. morphology
- b. color and pleochroism
- c. refractive indexes
- d. birefringence
- e. extinction characteristics
- f. sign of elongation

Table 5 lists the optical properties for a variety of fibrous constituents encountered in the analysis of building and insulation products. Table 7 presents a flow chart for the qualitative analysis of some of these materials. Central stop dispersion staining colors are listed in Table 6. It must be remembered that natural geological variations of asbestiform mineral deposits will produce exceptions to the data in Tables 5 and 6, and differences from laboratory standards.

The prepared slide is scanned identifying asbestos fibers using the optical properties of morphology, refractive indices, color pleochroism, birefringence, extinction characteristics, sign of elongation and dispersion staining characteristics.

5.3.1 Pleochroism

This is a property exhibited by some colored anisotropic substances. When viewed by polarized light pleochroic crystals change color as they are rotated. Examine the fiber of interest in plane polarized light (i.e. polarized in, analyzer out), and observe any color changes which result as it is rotated through 360°.

5.3.2 Isotropic/ Anisotropic

With the polarizer and analyzer crossed (i.e., dark field) rotate either the slide or the stage and observe the fiber of interest. An isotropic particle will remain dark (essentially invisible against the dark background). Conversely, anisotropic particles will present an image, which appears to fade in and out of the background (at 90° intervals) as it is rotated.

5.3.3. Angle of Extinction

As mentioned in Section 5.3.2., any anisotropic crystal extinguishes four times, between crossed polars, during a complete rotation.

This extinction occurs when the directions of vibration of the slow and fast rays of the fiber coincide with those of the polarizer and analyzer. Extinction may be one of three types:

- 1) Parallel or straight, when the fiber extinguishes parallel to the vibration direction the analyzer or polarizer (Figure I).
- 2) Symmetrical, when in the extinction position the vibration direction of the analyzer and polarizer are parallel to the diagnosis of a rhombic cross-section through a crystal (Figure I).

3) Oblique of inclined, when the fiber extinguishes at an oblique angle to the vibration directions of the analyzer and polarizer. This angle is known as the extinction angle, which is usually determined in terms of the slow vibration direction of the crystal.

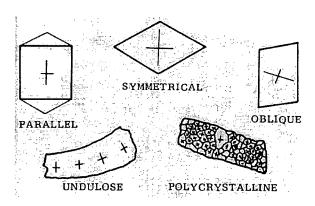


FIGURE I. Types of Extinction

5.3.4 Birefringence

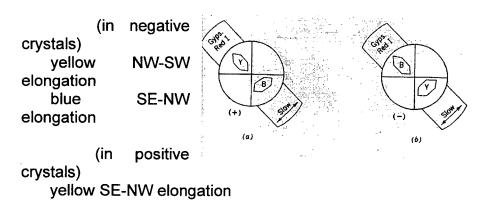
Birefringence (the difference between two indices of a particle on a given view) can be estimated from the interference colors observed when polarizers are crossed. As the stage (or slide) is rotated, isotropic particles (e.g., fibrous glass) will remain dark against the dark background. Particles with weak birefringence (e.g., quartz) will exhibit first order grays, whites, or yellows. As birefringence increases, higher order interference colors (reds, blues, greens, etc.) may be observed. As a rule, highly birefringent minerals appear brighter when rotated under crossed polarizers than do particles with weaker birefringence.

TABLE 1 - CATEGORIES OF BIREFRINGENCE STRENGTH WITH EXAMPLES

BIREFRINGENCE	INTERFERENCE COLOR IN SECTIONS 0.03 MM THICK	EXAMPLES, AND BIREFRINGENCE OF EXAMPLE	
Weak: 0.0.010	First order gray, white or yellow	Apatite: 0.0003	
Moderate: 0.010-0.025	First order red to second order green	Cancrinite: 0.0023-0.029	
Strong: 0.025-0.100	Upper second order into fifth order	Zircon: 0.062	
Very Strong: 0.100- 0.200	High order-sixth and higher	Calcite: 0.172	
Extreme: 0.200 and up	Very high order	Rutile: 0.285	

5.3.5 Sign of Elongation

Using a first-order red 1 plate and crossed polars determine the sign of elongation by positioning the fiber at an angle of 45° to the analyzer and/or polarizer. When the slow ray of the red plate is parallel to the elongation of fiber, and the interference color of the fiber is yellow, the mineral has a negative sign of elongation. Vice versa, if the interference color of the fiber is blue, the mineral has positive sign of elongation. In other words, the arrangement of colors:



blue NE-SW elongation

FIGURE II. Determination of Sign of Elongation

(a) positive elongation

(b) negative elongation

Excerpt from EMSLQASOPPLM.200.0 Sections for soil analysis

5.3.6 Dispersion Staining Colors

Dispersion staining is a technique for particle identification based on the difference between the dispersion of refractive index for a particle and the liquid medium in which the particle is immersed. In order to produce dispersion staining colors, the particle, and immersion liquid must have dispersion curves that intersect sharply in the visible light region. A special objective, containing annular and central stops in the back focal plane is required.

After isolating fibers of interest (sub-samples), follow the analysis flow chart (Table 7)

Note: Differences from standard characteristics may be observed due to natural variations in the conditions under which the minerals were formed and/or subjected to.

In the 1.55 HD refractive index oil, chrysotile will be readily identifiable from mineral wool or fiberglass by crossing the polars and using the 550-millimicron retardation plate to observe the colors of chrysotile. Both of the glass species are isotropic and will not show any colors. Many varieties of cellulose are close to 1.55 in index, but will not show chrysotile central dispersion staining colors. Characteristic magenta and blue colors identify chrysotile.

- a. If the fibers in the sample have a higher index of refraction than 1.55, have a negative sign of elongation, and appear blue by transmitted light, crocidolite is suspected. Prepare another slide with 1,700 refractive index oil. The color of crocidolite will be much bluer with an annular stop. The central stop dispersion staining colors are sometimes difficult to impossible to see because of the opacity of the dark blue fibers. If the fibers with the higher index than 1.55 are not blue, prepare a slide using 1.670 refractive index oil. Amosite has a positive sign of elongation and in the oil has central stop dispersion staining colors of yellow and magenta-blue.
- b. If the refractive index of the fibers is between 1.550 and 1.670 mount another preparation in 1.605 or 1.620 HD. The refractive indices for anthophyllite, tremolite, and actinolite vary naturally within the species. Anthophyllite can be distinguished from the other two by its parallel extinction. Actinolite has a light to dark green color with some pleochroism in transmitted light. The dispersion staining

Excerpt from EMSLQASOPPLM.200.0 Sections for soil analysis

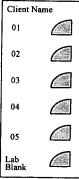
colors will have to be checked (i.e., actinolite DS colors in 1.63 RI oil are blue-magenta). A common interference mineral in this refractive index range is wollastonite. It also has a typical cleavage fragment morphology similar to the three asbestos minerals. Wollastonite has both a positive and a negative sign of elongation, parallel extinction and central stop dispersion stain colors in 1.605 HD of pale yellow and pale yellow to magenta. If further confirmation of wollastonite versus anthophyllite is

needed, wash a small portion of the sample in a drop of concentrated hydrochloric acid on a slide. Place the slide, with a coverslip in place, on a warm hot plate until dry. By capillary action, place 1.62 refractive index oil under the slipcover and then examine the slide. Wollastonite fibers will have a "cross-hatched" appearance across the length of the fibers and will not show central stop dispersion colors. Anthophyllite and tremolite will still show dispersion colors.

3.0 SAMPLE PREPARATION

3.1 MCE FILTERS

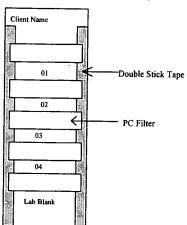
- Place the samples in an order corresponding to the clients COC.
- 2. Add a Laboratory Blank to the end of the series.
- 3. On a clean 1X3-microscope slide, scribe the billing number and the sample numbers with a diamond scribe.
- 4. Cut a wedge from the filter of each sample and place it on the slide above its ID number



- 5. Collapse the filters using the acetone vaporizer and fresh acetone.
- For AHERA, samples go to ASHING THE SAMPLE.
 All other analysis goes to CARBON COATING THE SAMPLES.

3.2 POLYCARBONATE FILTERS

- 1. Place the samples in an order corresponding to the clients COC.
- 2. Add a Laboratory Blank to the end of the series.
- 3. On a clean 1X3-microscope slide write the clients name and the sample numbers with a colored Sharpie permanent marker and cover with clear tape. This color will be used to designate that set of samples through analysis.
- 4. Using double stick tape, run a thin section down each side of the slides.
- 5. Cut rectangular sections of the polycarbonate filter and place on the slide so that each end adheres to the tape.
- 6. Go to CARBON COATING THE SAMPLES.



3.3 ASHING THE SAMPLES (MCE Filters Only)

SPI PLASMA PREP II LOW TEMPERATURE ASHER

- 1. Turn on the power strip to activate the pump, asher, and vacuum meter units, and turn on the oxygen
- 2. Load samples into the chamber.
- 3. Press the AC button.
- 4. Hold the chamber door closed and flip the vacuum switch up to the on position..
- 5. Wait until the chamber pressure is between 200 and 500 millitorr. button.
- 6. Flip the RF switch up to the on position
- 7. Adjust the Power and Tuning knobs to achieve a uniform plasma glow.
- 8. Adjust to a reading of three on the meter
- 9. Ash for the proscribed time

TO REMOVE SAMPLES:

- a. Turn off the RF switch.
- b. Turn off the vacuum switch.
- c. Allow the system to vent.

TO SHUT DOWN THE SYSTEM:

- a. Turn off the AC button.
- b. Turn off the power strip.
- c. Turn off the oxygen.

3.4 CARBON COATING THE SAMPLES

AIR VAX VACU-STATION CARBON COATER

TO START THE SYSTEM (MAKE SURE THAT ALL VALVES ARE CLOSED AND SWITCHES ARE OFF)

- 1. Turn on main power in rear of unit.
- 2. Press the [SYSTEM POWER] button.
- 3. Load samples to be coated, attach with double stick tape.
- Insert sharpened carbon rods.
- Press the [PUMP PROCESS] button.
- 6. Wait until the pressure reaches approx. $5x10^{-4}$ torr.

TO COAT A SAMPLE

- 1. Turn rotation control switch to on position.
- Turn on the Evaporation Control [POWER] button.
- Using the rheostat, slowly increase power until the carbon is just sparking, and continue until the carbon tip has been evaporated.
- Turn rheostat to zero.
- 5. Turn off the Evaporation Control [POWER] button.
- 6. Turn off rotation control.
- 7. Open toggle valve on left side of unit to vent the chamber.
- 8. Remove coated samples.
- 9. Turn off [SYSTEM POWER] button.
- 10. Turn off main power.

DENTON CARBON COATER

TO START THE SYSTEM (MAKE SURE THAT ALL VALVES ARE CLOSED AND SWITCHES ARE OFF)

- 1. Turn on main power.
- 2. Turn on cooling water.
- 3. Turn on mechanical pump.
- 4. Turn on diffusion pump.
- 5. Open backing valve.
- 6. Turn on thermocouple gauge, set to TC2 position, and turn on the high vacuum gauge.
- 7. Wait 15 minutes for diffusion pump to warm.

TO COAT A SAMPLE

- 1. Vent the chamber and lift off the bell jar.
- 2. Replace the used carbon rod with a new sharpened rod.
- 3. Place the sample in the coater affixing it to the metal plate with double stick tape.
- 4. Replace bell jar after checking for any debris around the rubber gasket.
- 5. Close the backing valve and the chamber vent.
- 6. Open the roughing valve.
- 7. Wait for the chamber pressure to reach below 50 mtorr.
- 8. Close the roughing valve.
- 9. Open the backing valve, then the main valve.
- 10. Set the high vacuum gauge range to the 10⁻⁴ range. IF THE RED LIGHT DOES NOT REMAIN ON WAIT TWO MINUTES AND TRY AGAIN.
- 11. Once the gauge drops below 1.0×10^{-4} switch the range to 10^{-5} and wait for the needle to reach 3×10^{-5} .
- 12. Turn on rotary power.
- 13. Turn on fill/glow power.
- 14. Slowly increase power until the carbon is just sparking, and continue until the carbon tip has been evaporated.
- 15. Turn off the fill/glow power.
- 16. Turn off rotary power.
- 17. Close the main valve.
- 18. Open the chamber vent and lift off the bell jar after venting is complete.

TO SHUT DOWN THE SYSTEM

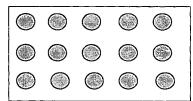
- 1. Replace the bell jar.
- 2. Turn off the diffusion pump.
- 3. Close the backing valve and the chamber vent.
- 4. Open the roughing valve and pump the chamber to below 100 mtorr.
- 5. Close the roughing valve and open the backing valve.
- 6. Wait 15 minutes for the diffusion pump to cool.
- 7. Close all vents and turn off all gauges.
- 8. Turn off the mechanical pump.
- 9. Turn off the system power.
- 10. Open the mechanical pump valve to vent pump.

NOTE: WHEN SYSTEM IS OFF ALL VALVES, VENTS AND SWITCHES SHOULD BE TURNED OFF.

3.5 DISSOLVING THE FILTER

3.5a THE JAFFE WICK

- Place a piece of cut Kimwipe onto the metal mesh screen in the petri dish making sure that the level of the solvent rises to touch the underside of the paper. For MCE filters use acetone and to dissolve PC filters use chloroform. (Optional)DMF and DMSO may replace acetone and in many cases will yield a better preparation.
- 2. Place three grids per sample in order on top of the Kimwipe making sure that the dull side of the grid is up.
- 3. Cut the collapsed coated sample filters in a grid pattern and carefully, using clean forceps, peal up one square at a time and place it onto one of the copper grids, carbon side up. (Carbon side down for NIOSH 7402)



Setup as shown

- 4. Replace the lid and label.
- 5. Allow to stand for at least 30 minutes for MCE and 60 minutes for PC. If time allows, leave the samples in the solution for one to two hours.
- 6. Pull from the solvent bath and allow drying for a minute before storing into a grid box.

3.5b THE CONDENSATION WASHER: (Optional)

- 1. Place a small piece of tissue paper onto a small piece of screen.
- 2. Place the screen into a petri dish with acetone for MCE or chloroform for PC. Similar to the Jaffe Wick.
- 3. Put your grids onto the damp tissue paper.
- 4. Carefully place your sample onto the grid.
- After the solvent has been brought to its boiling point, remove the cold finger from the condensation washer.
 Make sure the condensation washer is filled with acetone for MCE and chloroform for PC filters.
- 6. Carefully lift the tissue paper and screen off the wick and place onto the cold finger.
- 7. Replace the cold finger into the condensation chamber.
- 8. Wait at least five minutes then remove the cold finger.
- 9. Remove the screen and replace the cold finger.

- 10. Allow drying.
- 11. Mount samples on clip.

3.6 GRID STORAGE

- Grids are attached to an asymmetric copper clip with pre-cut sections of carbon double-stick tape. The clips are rectangular with a rectangular elongate opening running along the long axis of the clip. One end has a semi-circular notch on one end.
- Six grids fit on each clip and are arranged in a standard sequence. Counting away from the notch, the first and second grids are from sample one, the third and fourth from the second sample, and the fifth and sixth from the third sample. On the second clip are samples four in the first and second grid positions, five in the third and fourth positions and the lab blank in positions five and six. In the event of additional samples, continue this sequence, placing the blank at the appropriate end position. The notched end of the clip is oriented closest to the tip of the specimen arm,
- 3. Clips are lettered A through U inclusive, and placed in a specially designed holding box.
- 4. Grid boxes are uniquely and sequentially numbered
- 5. Included inside the box is a numbered inventory sheet with client name and billing number for tracking.
- 6. All unanalyzed grids (inclusive of the third prepped grid) are placed into standard numbered grid boxes, recorded on the grid box log sheet, and stored for three years.

4.0 SPECIALIZED SAMPLE PREPARATION

4.1 WATER SAMPLES-Method 100.1

- 1. Place the samples into the order noted on the COC.
- 2. Label a petri dish for each sample or for each dilution used per sample.
- 3. Setup the filtration apparatus, vertical walled fritted glass or disposable plastic units, with $0.22\mu m$ MCE filters and run a 100ml blank prior to sample filtering.
- 4. Thoroughly mix the samples by placing in a low temperature sonicator for 15 minutes and vigorously shake before removing the aliquot to filter.
- 5. Filter an appropriate amount or a series of aliquots and place the filter in the respective labeled petri dishes.
 For potable water filter, recommended amounts are 50ml & 100ml.
 For other water filter recommended amounts are 5ml, 10ml, & 25ml.
 Choosing the proper volume to filter comes with practice. If the filter exhibits discoloration (brown-tan), filter a smaller amount(s). If the aliquot is <50ml bring the sample to a volume of >50 using particle free water.
- 6. Allow the sample filters to dry. A heat lamp may be used to shorten the drying time of MCE filters only. *Do not subject the PC filters to heat.*
- 7. SÉE THE PROCEDURES UNDER AIR SAMPLE PREP, FOLLOWING THIS ORDER:

FOR MCE FILTERS

- a. Collapse & plasma ash the filter.
- b. Carbon coat the samples.
- c. Allow samples to soak in the Jaffe Wick for 1 hour or more.
- d. Remove grids and place on labeled carbon clip.

FOR PC FILTERS

- a. Cut and tape the filters to a clean glass slide.
- b. Carbon coat the samples.
- c. Allow samples to soak in the Jaffe Wick for a minimum of 12 hrs or longer to achieve proper clearing.
- d. Remove grids and place on labeled carbon clip.

8. Quality Control

- a. Prepare a Laboratory Blank with each sample set.
- b. If the set contains three or more samples, perform a Duplicate Prep on the first sample of each set.
- c. Sample Container Contamination Check
 - -one bottle in each batch or one bottle in 24.
 - -use a pre-washed bottle with 800 mls. of fiber free water

d. Record refrigerator temperature daily with NIST traceable thermometer. Temperature is to be maintained at $4^{\circ}C \pm 2^{\circ}C$.

5.0 SAMPLE ANALYSIS

DEFINITIONS:

Working Magnification: The magnification at which analysis should be performed.

<u>Fiber Criteria</u>: The attributes, size and shape, that a structure must display in order to be counted.

<u>Sizing of Fibers</u>: How the size of a structure needs to be recorded on the worksheet.

For AHERA, a check is placed in a column according to a structure being < or $\ge 5\mu m$ length. For EPA Level II, the actual length and width will need to be recorded.

Required EDX: The frequency at which structures need to be analyzed by EDX. For AHERA, each structure, which will cause the sample to exceed 70str/mm² will need EDX analysis. In the case of AHERA samples, this usually equates to the first four structures.

Required Diffraction Patterns: The frequency at which structures need to be analyzed by SAED.

Stopping Rules: The criteria required before analysis can be suspended.

Required Analytical Sensitivity: The criteria required for the number of grid openings to be analyzed. AHERA requires an A.S. of 0.005str/cc so a sample with 1200 liters being analyzed on 0.0129 mm² grid opening will require 5 openings for analysis.

<u>Laboratory Blanks</u>: A blank filter supplied by the lab, which is prepped along side of the samples to test for contamination. This section states if a laboratory blank is required and if the blank will need to be analyzed at the time of sample analysis.

Required Filters: The type of filter that the sample is to be taken on or filtered through.

<u>Pass / Fail Limit</u>: The level or concentration at which a sample or set of samples is past acceptable limits.

<u>Quality Control</u>: The steps to follow for QC. (S= Intra Analyst; D= Inter Analyst; SR=Intra Analyst Reprep, DRI=Inter Analyst Reprep, Inter Laboratory; V= Verified, B=Blanks).

PROCEDURES

- 1. Remove the first sample grid from the box and insert it into the TEM.
- 2. Bring the TEM to a magnification of 300 to 500x and inspect the grids to determine if at least 50% if the grid openings are intact. If two of the three grids are not 50% intact then the samples will have to be repreped.
- 3. Two grids are analyzed per sample. Separation of grid openings for each grid is recorded on the bench sheet with a one or two indicating the grid number and a line separating the two.

5.1 SAMPLE PREPARATION ACCEPTANCE

- 1. More than 50% of the grid must be covered by the replica.
- 2. Grids must have at least 50% intact grid openings
- 3. Grids must not have more than 10% opaque area due to incomplete filter dissolution.
- 4. Total Grid openings must have <50% overlapping of folded replica film
- 5. At least 20 grid openings with <5% holes and <5% opaque area due to incomplete filter dissolution.
- 6. Grid openings analyzed must not have rips or overlapping folds

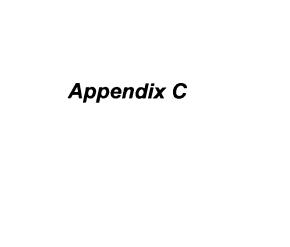
5.5 WATER 100.1

- 1. At a magnification of 300 to 500x, orient an intact grid opening on the middle of the screen.
- 2. Increase magnification to 10,000x taking care to remain in the chosen grid opening.
- 3. Log the grid opening number on the sample worksheet.
- 4. Move to the upper left corner of the grid square and begin traversing the grid using only one directional control. Once the opposite grid bar has been reached move over one screen width and traverse to the original grid bar. Take care not to count any structure twice or to miss any area of the grid opening.
- 4. If no fibrous structures have been located, repeat steps one through four until the stopping procedures have been reached.
- 5. If a fibrous structure has been located, check its morphology against the morphologies for each asbestos type. If morphology is consistent with asbestiform fibers, proceed to EDX analysis.
- 6. Obtain an EDX of the sample by following the procedures outlined in ENERGY DISPERSIVE X-RAY ANALYSIS and compare it to the spectra

for the asbestos types. Record the information on the sample worksheet. If the chemistry is consistent with one of the asbestiform minerals, proceed to Diffraction analysis.

- Obtain a diffraction pattern for the sample by following the procedures outlined in DIFFRACTION ANALYSIS. Record diffraction information on the sample worksheet.
- 8. If the morphology, diffraction, and EDX information match that of an asbestos type, then record the structure as asbestos.
- 9. For asbestos structures note whether it is a fiber, bundle, cluster, or matrix.
 - Fiber: a single fibril not connected to any non-fibrous debris.
 - Bundle: any group of three or more fibers lying parallel and with less than a single fiber diameter between them.
 - Cluster: a group of three or more fibers or bundles randomly oriented with three or more intersections.
 - *Matrix:* a bundle or fiber with one end free and the other end covered by or imbedded in a matrix material.
- 10. Working magnification is 10,000x.
- 11. Fiber criteria
 - Aspect ratio of 3:1
 - Must be ≥0.5μm length
 - Fibers intersecting top or left grid bars are recorded as twice the observed length.
 - Fibers intersecting the bottom or right grid bars are not counted.
- 12. Sizing of fibers
 - Record length and width of all counted asbestos fibers.
- 13. Required EDX
 - All structures
- 14. Required diffraction patterns
 - All structures
- 15. Stopping rules
 - May stop upon the completion of the 10th GO or the 100th structure, whichever comes first.
- 16. Required detection limit
 - -N/A
- 17. Laboratory blanks
 - 10% of samples submitted
 - Prep blanks with 100ml particle free water and analyze to a D.L. of 0.05
- 18. Sample Container Contamination Check
 - One bottle in each batch or one bottle in 24.
 - Use pre-washed bottle with 800 mls. of fiber free water
- 19. Required filters
 - 0.22 MCE
- 20. Pass / Fail limit

- N/A
- 21. Quality Control
 - Analyze Laboratory Blanks accompanying sample sets.
 - Analyze Replicate preps accompanying sample sets.
 - Perform contamination checks on sample containers
 - 10% QC is required for all water samples submitted. Duplicates are prepared and analyzed for 1 in 15 samples. Replicates are prepared and analyzed for 1 in 50 samples. Lab blanks are prepared and analyzed for 1 in 20 samples.



STANDARD OPERATING PROCEDURE FOR THE DATA VALIDATION OF ASBESTOS ANALYTICAL DATA

This document presents specific data validation requirements for asbestos analytical data analyzed by U.S. EPA <u>Test Method for the Determination of Asbestos in Bulk Building</u> materials, EPA/600/R-63/116, July 1993.

Case Narrative

A case narrative will be included with each data package and should be reviewed for information specific to the associated data such as abnormalities encountered with the samples, reanalyses, and deviations from the referenced analytical method.

Blanks Laboratory Duplicates

Verify that the duplicate samples met the following criteria and that the laboratory provided the following information:

- Verify that the laboratory has conducted a duplicate analysis sample at a frequency of 5% (one in twenty samples).
- Verify that the duplicate analysis was prepared and analyzed at the same time, using the same procedure as the associated samples.
- Verify that the duplicate sample analysis result is similar to the sample result (+/25%). If the difference between the sample and the duplicate is greater than 25%
 qualify both sample results estimated "J".
- If a duplicate was not analyzed for each batch of 20 or less samples, qualify all associated samples estimated "J".

The blank data results are reviewed to assess the extent of contamination introduced through sampling, sample preparation and analysis. Summarize all blank results in the validation narrative.

Precision

The review of field and laboratory precision provides information on the laboratory reproducibility and whether sampling activities are adequate to acquire consistent samples. Field Blanks should not be used for laboratory duplicates.

Laboratory Duplicates

Verify that the laboratory duplicate samples have met the following criteria and that the laboratory provided the following information:

 Verify that a field duplicate was collected and analyzed for every 20 or less field samples collected.

Verify that the laboratory duplicate analysis was prepared and analyzed at the same tim, using the same procedure as the associated samples.

- Verify that the duplicate sample analysis result is similar to the sample result (+/25%). If the difference between the sample and the duplicate is greater than 25%
 qualify both sample results estimated "J".
- If a duplicate was not analyzed for each 20 or less field samples, qualify all associated sample data estimated "J".

Field Duplicates

Verify that the field duplicate samples met the following criteria and that the laboratory provided the following information:

- Verify that a field duplicate was collected and analyzed for every 20 or less field samples collected.
- Verify that the field duplicate analysis was prepared and analyzed at the same time, using the same procedure as the associated samples.
- Verify that the field duplicate sample analysis result is similar to the sample result (+/-25%). If the difference between the sample and the duplicate is greater than 25% qualify both sample results estimated "J".
- If a field duplicate was not collected and analyzed for each batch of 20 field samples, qualify all associated sample data estimated "J".

Holding Times

Verify that all samples were within 180 days of collection. If holding times are exceeded qualify sample results as follows"

- If holding times are >180 days but <360 days qualify all associated data estimated "J".
- If holding times are >360 days, reject "R" all associated data

Overall Assessment and Summary

Summarize the qualified results as specified in the case narrative.